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HOLDOVER TIME FIELD TEST SUBSTITUTION

by

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16. Abstract

The objectives of this project were to develop a set of tests that will lead to the creation of a holdover time table for a candidate fluid under all the types of precipitation included in such a table, and to prepare detailed laboratory test procedures.

The set of tests required was defined at a November 1997 meeting of an SAE workgroup, which decided to include all types of precipitation and to identify each test by a set of environmental parameters that include precipitation type, icing intensity, and air temperature. In most cases, two icing intensities, a moderate and a high value, were associated with each precipitation type and each air temperature value.

A tolerance on both test parameters – icing intensity and air temperature – was defined to ensure that all tests could be performed in the same way, in different facilities, at different times, by different people, and still achieve good reproducibility.

Calibration and fluid tests were conducted to evaluate the procedures. Following these tests and an SAE G-12 Fluids Subcommittee meeting in March 1999, modifications were made and the resulting procedures were presented for an SAE ballot.

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16. Résumé

Ce projet avait pour objectifs de définir une série d'essais devant déboucher sur la création de la table des durées d'efficacité d'un fluide candidat, sous tous les types de précipitations prévues par cette table, et de mettre au point des protocoles détaillés d'essais en laboratoire.

La série d'essais nécessaires a été définie lors d'une réunion, en novembre 1997, d'un groupe de travail de la SAE. Celui-ci a alors décidé d'englober tous les types de précipitations et de définir chaque essai en fonction d'une gamme de paramètres environnementaux, dont le type de précipitations, l'intensité de givrage et la température de l'air. La plupart des essais mettent en jeu deux intensités de givrage, modérée et élevée, pour chaque type de précipitations et chaque température d'air retenus.

Une tolérance a été admise pour les paramètres d'intensité de givrage et de température de l'air afin que les essais, réalisés selon la même méthode mais dans des laboratoires différents, à des époques différentes et par des opérateurs différents, offrent une bonne reproductibilité.

Des essais d'étalonnage ont d'abord été réalisés, puis des essais des fluides comme tels, qui ont servi à évaluer les protocoles. Par suite de ces essais et d'une réunion du sous-comité G-12 de la SAE sur les durées d'efficacité, tenue en mars 1999, des modifications ont été apportées aux protocoles et ceux-ci ont été soumis au scrutin de la SAE.

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SUMMARY

Deicing and anti-icing fluids are commonly used in winter to remove and prevent aircraft contamination by frozen deposit. Anti-icing fluids protect the aircraft for a time period that depends on environmental conditions, including the nature of precipitation, the temperature, and the precipitation intensity. To help the pilot and the airport management to assess this protection time, the SAE publishes tables that address several types of precipitation and present fluid holdover time guidelines as part as of the ARP4737 standard. The Holdover Time SAE G-12 Subcommittee is in charge of establishing and updating these guidelines.

At a meeting in Atlanta on October 1 and 2, 1997, the Fluids G-12 Subcommittee recommended the creation of a workgroup to examine criteria associated with laboratory testing of fluids. The Workgroup on Laboratory Methods to Derive Holdover Time Guidelines met in Montreal on November 20 and 21, 1997 and recommended that the Anti-icing Materials International Laboratory (AMIL) write detailed laboratory test procedures based on the resolutions of the workgroup.

The objectives of this project were:

- to establish a set of tests that will lead to the creation of a holdover time table for candidate fluids under conditions of frost, freezing drizzle, light freezing rain, freezing fog, snow and rain on a cold-soaked wing, at various temperatures and icing intensities; and
- to document test procedures for each type of test to be presented for a SAE ballot.

The set of tests required to develop a holdover time table was defined at the November 1997 meeting. It must include each type of precipitation mentioned above and cover the overall range of temperatures. The air temperature values for the tests are 0, -3, -14, -25°C, and the lowest operational use temperature (LOUT) for frost; -3, -14, -25°C and LOUT for freezing fog and snow; -3 and -10°C for freezing drizzle and light freezing rain; and 1°C for rain on a cold-soaked wing. For frost and for all tests performed at the LOUT, fluids are tested at only one icing intensity at each temperature; but for all other

types of precipitation and for temperatures other than the LOUT, fluids are tested at two icing intensities: moderate and high. The former yields the longer time of the corresponding cell in the holdover time table and the latter yields the shorter time of the same cell. The icing intensity varies for each type of precipitation.

The procedures were based on the anti-icing endurance performance tests described in AMS1428C and modified to take into account some characteristics of the process used to substantiate and generate the current holdover time tables.

Tolerances on the icing intensity and the air temperature test parameters were defined to ensure that all tests can be performed in the same way in different facilities, at different times, by different people, and still achieve a good test reproducibility.

All tests were conducted in climatic chambers with specialized equipment. Different types of liquid precipitation require different nozzle sizes. Freezing fog requires a humidification system and a frosticator able to maintain the test panels at a temperature 3°C lower than the air temperature. A cold soak box is used for rain on a cold-soaked wing simulation. A snow generation system was specially designed and built for the snow tests. It consists of an automated distribution mechanism capable of evenly applying snow over a test panel. The artificial snow consists of agglomerates of tiny (about 100 µm) frozen droplets.

For all tests but frost, the fluid test panels are 500 mm long, 300 mm wide, and 3.2 mm thick. Their temperature is the same as the air at the beginning of the test, but is allowed vary during the test. For frost, the panels are 300 mm long, 100 mm wide, and 1.6 mm thick, and are maintained at a prescribed temperature throughout the test by a special cooling system.

The procedures call for calibration and fluid tests. Calibration tests are performed to measure the average icing intensity and its distribution over each test panel. For each 500 mm x 300 mm panel at least twelve 100 x 100 mm pans or plates are used to collect the ice, and for each 300 mm x 100 mm plate, three plates are used for the same purpose. The average weight and standard deviation of the ice collected by the calibration pans or plates allow the calculation of the average and standard deviation of icing intensity. As a

rule, the average icing intensity and the standard deviation must remain respectively within 4 and 6 percent of the target intensity. The tolerance is increased when the target intensity is small.

The tolerance on average air temperature is usually ± 0.5°C, except at -25°C and at the LOUT, when it is increased to 2.0°C. The tolerance on the air temperature standard deviation is usually 0.2 or 0.3°C, except at -25°C and at LOUT, when it is increased to 0.5°C.

Over four hundred tests, including calibration and fluid tests, were conducted under all types of precipitation. The tests verified that the parameter tolerances could be easily met. The fluid tests also identified problems in the test procedures, such as lack of reproducibility due to variations in fluid texture at the time of application. A March 1999 meeting led to a decision to modify the test fluid application procedure and to adopt that of the current AMS1428C anti-icing endurance tests. Snow tests were performed with a standard fluid, fluid X, formulated by Union Carbide Corporation, to establish a reference to snow generation system calibration.

As a follow-up to this work, a procedure for each precipitation type of was written and proposed for a SAE ballot. At the SAE March 1999 meeting, the results of the ballot were discussed and the procedures amended. The modified procedures, which were again submitted for ballot in May 1999, are included as appendices to this report.

SOMMAIRE

Des fluides dégivrants et antigivre sont couramment utilisés en hiver pour débarrasser les aéronefs de dépôts glacés et prévenir leur contamination sous précipitations givrantes. Les fluides antigivre protègent les aéronefs pendant une période qui dépend de diverses conditions environnementales, dont la nature de la précipitation, la température et l'intensité de la précipitation. Pour aider les pilotes et la direction des aéroports à évaluer la durée de la protection, la SAE publie des tables de durée d'efficacité des fluides, incorporées à la norme ARP4737. Ces tables prennent en compte plusieurs types de précipitations. Le sous-comité G-12 de la SAE sur les durées d'efficacité est chargé d'établir et de mettre à jour ces tables.

À une réunion tenue à Atlanta les 1er et 2 octobre 1997, le sous-comité G-12 avait recommandé la création d'un groupe de travail chargé d'examiner les critères à appliquer aux essais en laboratoire des fluides antigivre. Le groupe de travail, qui s'est réuni à Montréal les 20 et 21 novembre 1997, a recommandé que le Laboratoire international des matériaux antigivre (LIMA) élabore les protocoles détaillés d'essais fondés sur les résolutions adoptées à la réunion du groupe.

Ce projet avait pour objectifs :

- de définir une série d'essais devant déboucher sur la création de tables de durées d'efficacité pour les fluides candidats dans des conditions de givre, de bruine verglaçante, de faible pluie verglaçante, de brouillard givrant, de neige et de pluie sur une aile sur-refroidie, à diverses températures et selon plusieurs intensités de givrage ;
- de documenter les protocoles d'essais pour chaque type d'essai, à soumettre au scrutin de la SAE.

La série d'essais nécessaires pour élaborer une table de durées d'efficacité a été définie au cours de la réunion de novembre 1997. Cette série doit comprendre tous les types de précipitations ci-dessus et couvrir un large éventail de températures. Les valeurs de température de l'air à inclure dans les essais ont été établies à 0, -3, -14, -25°C et à la limite inférieure de température d'utilisation (LITU), pour le givre; à -3, -14, -25°C et à la

LITU, pour le brouillard givrant et la neige ; à -3 et -10°C, pour la bruine verglaçante et la faible pluie verglaçante ; et à 1°C pour la pluie sur une aile sur-refroidie. Pour les essais simulant des conditions de givre et tous les essais à la LITU, une seule intensité de givrage est étudiée, à chaque température ; mais pour tous les autres types de précipitations et à toutes les autres températures autres que la LITU, deux intensités de givrage, modérée et élevée, sont mises en jeu. Pour chaque type de précipitation et à chaque température, la durée d'efficacité des fluides est plus longues sous une intensité de givrage modérée que sous une intensité de givrage élevée. Les valeurs rattachées à l'intensité de givrage varient selon le type de précipitation.

Les protocoles ont été élaborés d'après les épreuves de performance antigivre décrits dans la norme AMS1428C, sous réserve de quelques modifications pour tenir compte de certaines caractéristiques de la méthode utilisée pour justifier et générer les tables de durée d'efficacité actuelles.

Des tolérances ont été admises pour les paramètres d'intensité de givrage et de température de l'air afin que les essais, réalisés selon la même méthode mais dans des laboratoires différents. à des époques différentes et par des opérateurs différents, offrent une bonne reproductibilité.

Tous les essais ont eu lieu dans des chambres climatiques, à l'aide de matériel spécialisé. Des buses de calibres différents doivent être utilisées pour simuler les différents types de précipitations liquides. Quant au brouillard givrant, il nécessite la mise en œuvre d'un système d'humidification et de refroidissement capable de maintenir les panneaux d'essai à une température inférieure de 3 degrés Celsius à la température de l'air. Une boîte refroidie est utilisée pour les essais de pluie sur une aile sur-refroidie. Un système de saupoudrage de neige a été spécialement conçu et construit pour les essais sous précipitations neigeuses. Il s'agit d'un dispositif de distribution automatisé capable de répartir uniformément la neige sur un panneau d'essai. La neige artificielle est constituée d'agglomérats de minuscules (environ 100 μm) gouttelettes d'eau congelées.

Pour tous les essais, sauf les essais sous givre, les panneaux d'essai mesurent 500 mm de longueur, 300 mm de largeur et 3,2 mm d'épaisseur. Au début de l'essai, leur température

est la même que celle de l'air, mais on la laisse varier par la suite. Les panneaux utilisés pour les essais sous givre mesurent 300 mm de longueur, 100 mm de largeur et 1,6 mm d'épaisseur et sont maintenus à nue température prescrite pendant toute la durée de l'essai par un système de refroidissement spécial.

Les protocoles exigent des essais d'étalonnage préalables aux essais des fluides comme tels. Les essais d'étalonnage ont pour objet de mesurer l'intensité de givrage moyenne et la distribution du dépôt glacé sur chaque panneau d'essai. Dans le cas des panneaux de 500 mm x 300 mm, les dépôts sont recueillis dans au moins douze bacs ou plaques de 100 mm x 100 mm ; dans le cas des panneaux de 300 mm x 100 mm, trois plaques sont utilisées. La moyenne et l'écart-type des quantités de glace ainsi recueillis permettent de calculer la moyenne et l'écart-type de l'intensité de givrage. Règle générale, la moyenne et l'écart-type de l'intensité de givrage doivent s'écarter d'au plus 4 et 6 p. cent, respectivement, de l'intensité visée. La tolérance admise est d'autant plus grande que l'intensité visée est faible.

La tolérance admise pour la température moyenne de l'air est habituellement de ± 0,5°C, sauf à -25°C et à la LITU, où elle est de 2,0°C. La tolérance pour ce qui est de l'écart-type de la température de l'air est habituellement de 0,2 ou 0,3°C, sauf à -25°C et à la LITU, où elle est de 0,5°C.

Plus de quatre cents essais, essais d'étalonnage et essais des fluides compris, ont été réalisé sous tous les types de précipitations. Les essais ont confirmé que les tolérances relatives aux paramètres d'essai sont réalistes : elles sont rarement dépassées. Les mêmes essais ont mis au jour des problèmes de reproductibilité attribuables aux variations de la texture des fluides au moment de l'application. Lors d'une réunion tenue en mars 1999, il a été décidé de changer la méthode d'application des fluides d'essai pour celle actuellement utilisée pour les essais de performance antigivre de la norme AMS1428C. Les essais sous neige ont été réalisés à l'aide d'un fluide standard, le fluide X, formulé par Union Carbide Corporation, afin d'avoir une valeur de référence pour l'étalonnage du simulateur de précipitations neigeuses.

Par suite de ces travaux, une protocole d'essai pour chaque type de précipitation a été élaboré et proposé à la SAE pour mise au scrutin. À la réunion de mars 1999 de la SAE, les résultats de scrutin ont été discutés et les protocoles, modifiés. Ces derniers, qui ont été de nouveau soumis au scrutin en mai 1999, sont annexés au présent rapports.

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1. INTRODUCTION

Ongoing development in ground aircraft deicing and anti-icing techniques is supervised by the SAE G-12 Holdover Time Subcommittee, which is in charge of establishing and updating the fluid holdover time guidelines used in operation and currently presented in the ARP4737 standard.

In December 1990, Transport Canada and the Federal Aviation Administration (FAA) undertook the task of substantiating the existing guideline tables for Type I and Type II fluids. A specific test procedure was defined for snowfall conditions, based on a correlation established between real aircraft contamination and aluminum plate contamination. During the first seven winters of testing, the Type I table and particularly the Type II table have been modified when necessary to account for new results, though the tests were mostly performed with the same fluids.

At the end of 1993, the first of a new generation of fluids, the Type IV fluids, was commercialized. This new fluid was characterized by an extended ice holdover protection compared to the existing Type II fluids, with its certification water spray endurance time two and a half times longer than that of the Type II fluids. It was then necessary to create a special table for this new type of fluid. However, while most of the commercial Type II fluids formulations have remained virtually unchanged for several years, the Type IV fluid formulations are modified almost every year by the fluid manufacturers to improve their overall performance.

The objective of Transport Canada and the FAA, which was first limited to the substantiation of existing tables, then became an endless race to keep up with fluid development. When the priorities of both agencies shifted toward other issues, it became necessary to turn the burden of the fluid anti-icing performance evaluation back to the fluid manufacturers, while still maintaining solid confidence in the information provided. The G-12 Steering Committee decided then to evaluate the possibility of testing fluids entirely in laboratory.

At the Fluids G-12 Subcommittee meeting held in Atlanta on October 1 and 2, 1997, a recommendation was made to create a workgroup whose task was to look into criteria

and costs associated with laboratory testing of fluids to replace the current testing. The Workgroup on Laboratory Methods to Derive Holdover Time Guidelines met in Montreal on November 20 and 21, 1997, and recommended that the Anti-icing Materials International Laboratory (AMIL) write detailed laboratory test procedures based on the resolutions of the workgroup. This report presents these test procedures as well as the work conducted by AMIL at the Université du Québec à Chicoutimi, on which these procedures are partially based.

2. OBJECTIVES

The first objective of this work was to determine a set of tests which will permit the building of a holdover time guidelines table for a candidate aircraft de/anti-icing fluid under frost, freezing drizzle, light freezing rain, freezing fog, snow, and rain on a cold-soaked wing conditions. If these tests are to be used as part of a fluid certification, they must be described in detail and consequently, the testing parameters must also be well defined.

The second objective was to write the test procedures to be presented for an SAE ballot.

3. DETERMINATION OF THE SET OF TESTS

The determination of the set of tests was based on the first objective of the testing process, to determine the values in the holdover time guidelines table. As shown in Figure 1, a table consists of nine columns: the first two columns identify temperature ranges in degrees Celsius and Fahrenheit, the third column represents the dilutions, and the remaining columns represent six different types of precipitation: frost, freezing fog, snow, freezing drizzle, freezing rain, and rain on a cold-soaked wing. Therefore, the set of tests required to build a holdover time table must include each type of precipitation and cover the overall range of temperatures.

Each cell contains two numbers representing two different holdover times. It was decided at the Montreal meeting that:

- the shortest time would represent the holdover time obtained with a fluid tested in the most severe condition for this cell;
- the longest time would represent the holdover time obtained with a fluid tested in a moderate condition for this cell; and
- the most severe and moderate conditions would be determined by meteorological standards modified to take into account the likelihood of an icing intensity at a given temperature. These conditions are shown in Table 1. The temperature ranges currently used in the holdover time tables are delimited by 0, -3, -10 or -14, and -25°C, depending on the type of precipitation. The values selected to represent the most severe conditions are presented in bold, those representing the moderate conditions are in bold italic. For the frost condition, only one value is presented in each cell.

TABLE 4

SAE TYPE IV COMPOSITE FLUID HOLDOVER TABLE

Guideline for Holdover Times Anticipated for SAE Type IV Fluid Mixtures as a Function of Weather Conditions and OAT
THE RESPONSIBILITY FOR THE APPLICATION OF THESE DATA REMAINS WITH THE USER

OAT		SAE Type IV Fluid Concentration	Approximate Holdover Times Under Various Weather Conditions (hours : minutes)					
°C	°F	Neat - Fluid / Water (Vol% / Vol%)	*FROST	FREEZING FOG	SNOW	***FREEZING DRIZZLE	LIGHT FREEZING RAIN	RAIN ON COLD SOAKED WING
above 0°	above 32°	100/0	18:00	2:20 - 3:00	0:45 - 1:25	0:40 - 1:00	0:35 - 0:55	0:10 - 0:50
		75/25	6:00	1:05 - 2:00	0:20 - 0:40	0:30 - 1:00	0:15 - 0:30	0:05 - 0:35
		50/50	4:00	0:20 - 0:45	0:05 - 0:20	0:10 - 0:20	0:05 - 0:10	
0 to -3	32 to 27	100/0	12:00	2:20 - 3:00	0:35 - 1:00	0:40 - 1:00	0:35 - 0:55	
		75/25	5:00	1:05 - 2:00	0:20 - 0:35	0:30 - 1:00	0:15 - 0:30	
		50/50	3:00	0:20 - 0:45	0:05 - 0:15	0:10 - 0:20	0:05 - 0:10	
below -3 to -14	below 27 to 7	100/0	12:00	0:40 - 3:00	0:20 - 0:40	**0:30 - 1:00	**0:30 - 0:45	
		75/25	5:00	0:35 - 2:00	0:15 - 0:30	**0:30 - 1:00	0:15 - 0:30	
below -14 to -25	below 7 to -13	100/0	12:00	0:20 - 2:00	0:15 - 0:30			
below -25	below -13	100/0	SAE Type IV fluid may be used below -25° C (-13° F) provided the freezing point of the fluid is at least 7° C (13° F) below the OAT and the aerodynamic acceptance criteria are met . Consider use of SAE Type I when SAE Type IV fluid cannot be used.					

°C = Degrees Celsius
°F = Degrees Celsius
OAT = Outside Air Temperature
FP = Freezing Point
* During conditions that apply to aircraft protection for ACTIVE FROST.
** The lowest use temperature is limited to -10° C (14° F).
***Use light freezing rain holdover times if positive identification of freezing drizzle is not possible.

CAUTION: THE TIME OF PROTECTION WILL BE SHORTENED IN HEAVY WEATHER CONDITIONS, HEAVY PRECIPITATION RATES OR HIGH MOISTURE CONTENT. HIGH WIND VELOCITY OR JET BLAST MAY REDUCE HOLDOVER TIME BELOW THE LOWEST TIME STATED IN THE RANGE. HOLDOVER TIME MAY ALSO BE REDUCED WHEN AIRCRAFT SKIN TEMPERATURE IS LOWER THAN OAT. THE ONLY ACCEPTABLE DECISION CRITERIA TIME IS THE SHORTEST TIME WITHIN THE APPLICABLE HOLDOVER TIMETABLE CELL.

FLUIDS USED DURING GROUND DEICING ARE NOT INTENDED FOR AND DO NOT PROVIDE ICE PROTECTION DURING FLIGHT

(Transport Canada Air Carrier Advisory Circular 0113R, 1997)

Figure 1: Example of a Holdover Time Guidelines Table

Table 1: Chart of Precipitation Rates for Holdover Times

OAT		Precipitation Rates Under Various Weather Conditions, g/dm²/h					
°C	°F	Frost	Freezing Fog	Snow	Freezing Drizzle	Light Freezing Rain	Rain on a Cold-Soaked Wing
0	32	0.20	5 – *2*	25 - *10*	13 - *5*	25 - *13*	75 - *5*
-3	27	0.20	5 – *2*	25 - *10*	13 - *5*	25 - *13*	
-7	19	0.20	5 – *2*	25 - *10*	13 - *5*	25 - *13*	
-10	14	0.20	5 – *2*	25 - *10*	13 - *5*	25 - *13*	
-14	7	0.13	5 – *2*	25 - *10*			
-25	-13	0.05	5 – *2*	10 - *5*			
<-25	<-13	TBD*	5	5			

* To be determined

The set of tests to be performed for each type of precipitation is summarized in Table 2.

Table 2: Summary of the Test Conditions

Frost		Freezing Fog		Snow		Freezing Drizzle		Light Freezing Rain		Rain on a Cold-Soaked Wing	
T	I	T	I	T	I	T	I	T	I	T	I
0	0.20	-3	2	-3	10	-3	5	-3	13	1	5
-3	0.20	-3	5	-3	25	-3	13	-3	25	1	75
-14	0.13	-14	2	-14	10	-10	5	-10	13		
-25	0.05	-14	5	-14	25	-10	13	-10	25		
<-25	R	-25	2	-25	5						
		-25	5	-25	10						
		<-25	5	<-25	5						

T: Air temperature (°C) **I**: Icing intensity (g/dm²/h) **R**: To be reported

4. EQUIPMENT AND SIMULATION OF PRECIPITATION

4.1 Climatic Chambers

The anti-icing endurance tests were performed in various climatic chambers at the Université du Québec à Chicoutimi (UQAC). The choice of a chamber for each type of test was determined by its size: the freezing fog and frost tests were conducted in the smaller chambers and the freezing drizzle, light freezing rain, and rain on a cold-soaked wing tests in the 9 m high climatic chamber. The artificial snow was made in the 9 m high chamber and the actual snow tests were performed in a smaller chamber for convenience.

4.2 Simulation of Precipitation

For the supercooled precipitation, two different types of water spray system were used: one for the freezing fog tests and the other for the freezing drizzle, light freezing rain, and rain on a cold-soaked wing tests. All systems used ASTM D1193 Type IV water.

4.2.1 Freezing Fog Water Spray

The system used for the freezing fog tests is based on a pneumatic water spray nozzle oscillating over the test area. The nozzle, located 1.45 m above the test plate support, allows for the production of very fine droplets presenting a 22 ± 5 μm median volume diameter (see Figure 2 and Figure 3). The water spray is continuous.

4.2.2 Freezing Drizzle, Light Freezing Rain, and Rain on a Cold-Soaked Wing Water Spray

The system used for the freezing drizzle, light freezing rain, and rain on a cold-soaked wing tests is based on a hydraulic water spray nozzle oscillating over the test area. The nozzle, located about 7.0 m above the plate support, allows for the production of droplets presenting median volume diameters greater than 150 μm depending on the nozzle hole diameter. The water spray is controlled by an "on/off" pulse. The pulse ratio determines the intensity of

precipitation for a given nozzle. For the light freezing rain and the rain on a cold-soaked wing tests, the droplet median volume diameter is 970 μm (see Figure 4 and Figure 5).

For the freezing drizzle tests, the median volume diameter is 237 μm (see Figure 6 and Figure 7).

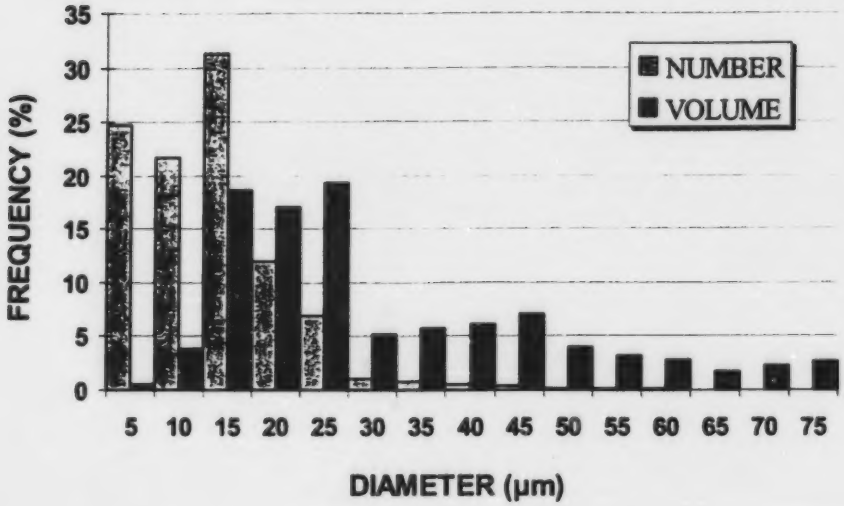


Figure 2: Freezing Fog Spraying Nozzle: Droplet Size Frequency

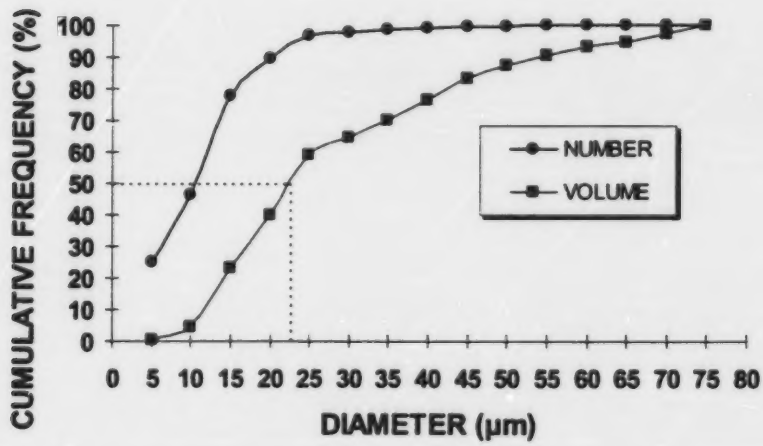


Figure 3: Freezing Fog Spraying Nozzle: Droplet Size Cumulative Frequency

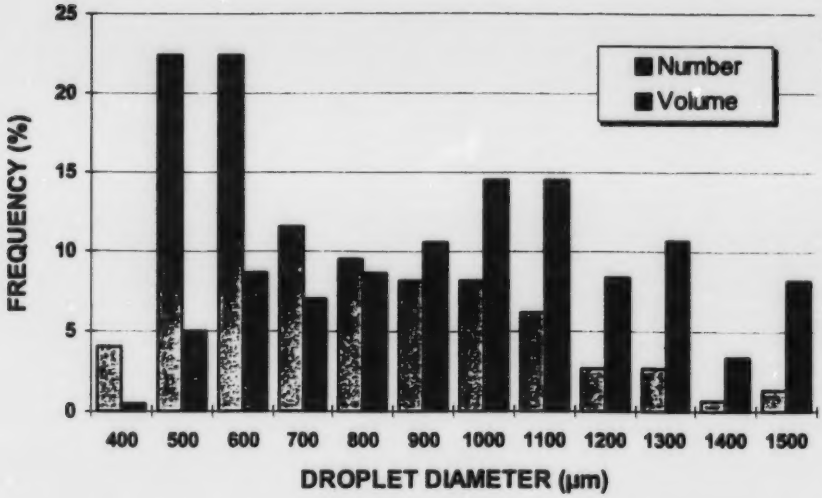


Figure 4: Light Freezing Rain and Rain on a Cold-Soaked Wing Spraying Nozzle: Droplet Size Frequency

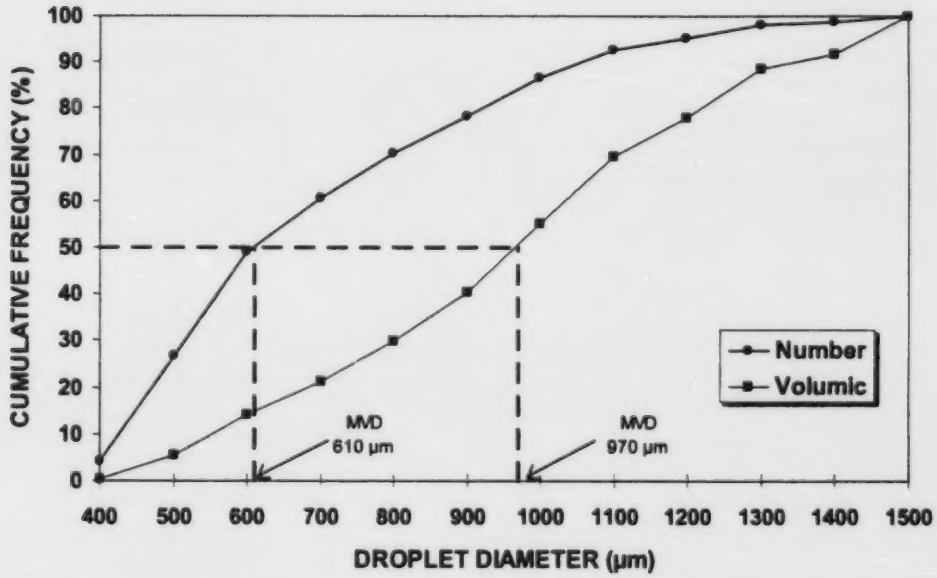


Figure 5: Light Freezing Rain and Rain on a Cold-Soaked Wing Spraying Nozzle: Droplet Cumulative Size Frequency

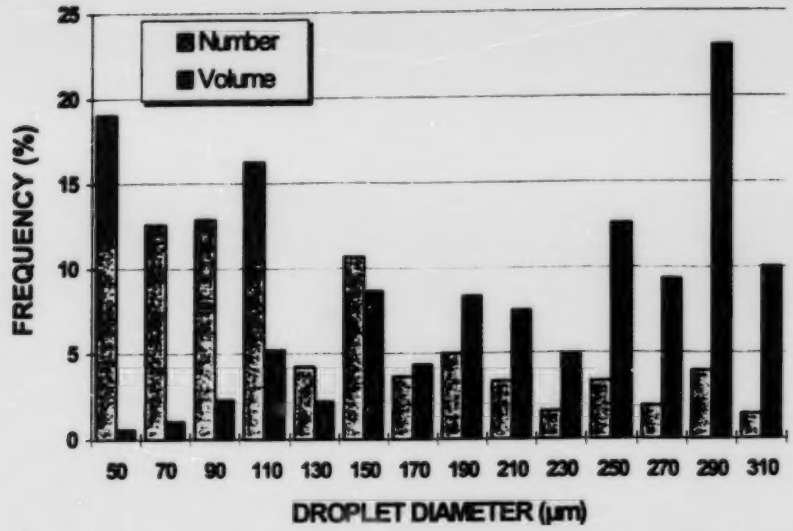


Figure 6: Freezing Drizzle Spraying Nozzle: Droplet Size Frequency

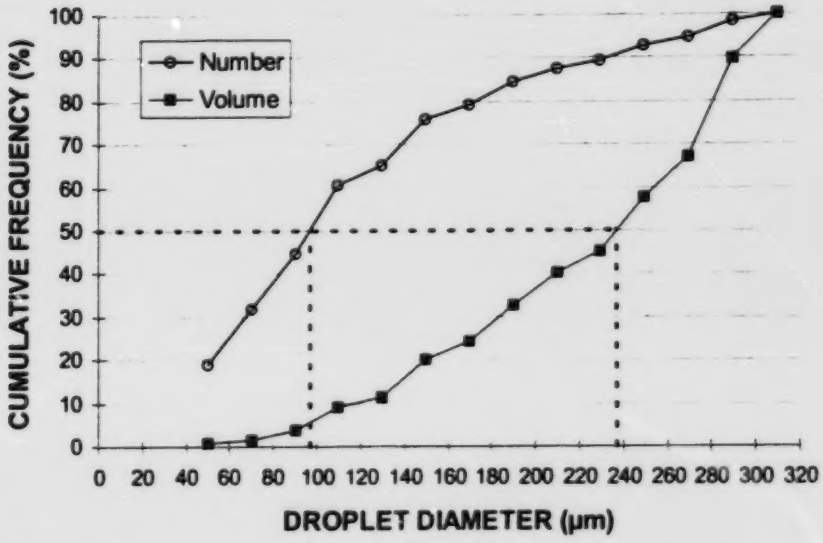


Figure 7: Freezing Drizzle Spraying Nozzle: Droplet Size Cumulative Frequency

4.2.3 Frost Generation

Frost is generated when a mass of humid air comes in contact with a surface colder than the air. The quantity of frost accumulated depends on the level of humidity in the air and the temperature differential between the air and the surface of deposition.

The UQAC frost generation system is contained in a cold chamber and includes:

- a frosticator, shown in Figure 8, which is a plate support on which a set of six plates inclined at a 10° angle is continuously maintained at a prescribed temperature cooler than that of the ambient air throughout the test by its own cooling system; and
- a humidifier, in which the volume and the temperature of the water are continuously controlled at prescribed levels throughout the test.

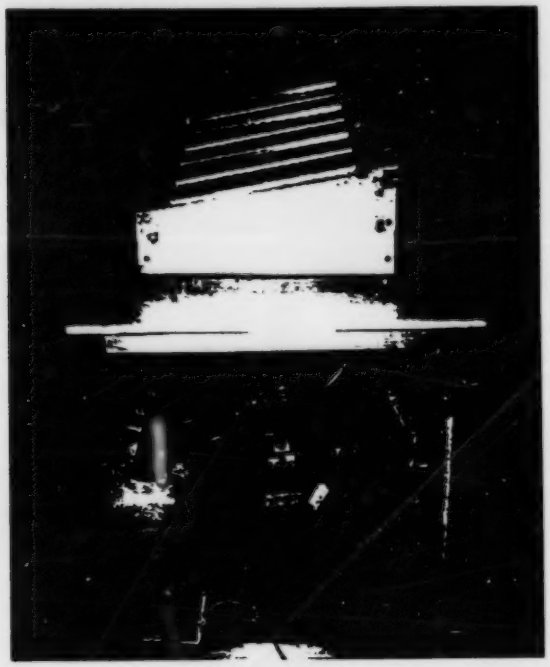


Figure 8: Frosticator

4.2.4 Snow Generation

Snow is generated by the accumulation of rime formed by freezing small water droplets produced with a fine spray hydraulic nozzle. Agglomerates of the frozen water droplets form instantaneously, trapping air in-between. The density of the artificial snow collected at this time was 0.25 ± 0.2 g/cm^3.

The snow can be stored for at least a week, but must be discarded if any sign of sintering, such as color change from white to clear, is visible.

For the snow test, the snow was placed into a special box and continuously mixed to prevent it from compacting. At the bottom of this box, a cylinder grooved with several rows of cavities rotated as directed by a computer program. When the cavities were on the top of the cylinder, they filled with snow, and when they were on the bottom of the cylinder, the snow fell down by gravity. The rotation speed of the cylinder determined the precipitation intensity, which was limited to 30 g/dm²/h. The bottom of the machine was located about 0.5 m above the test plates. A schematic of the snow distribution system is presented in Figure 9.

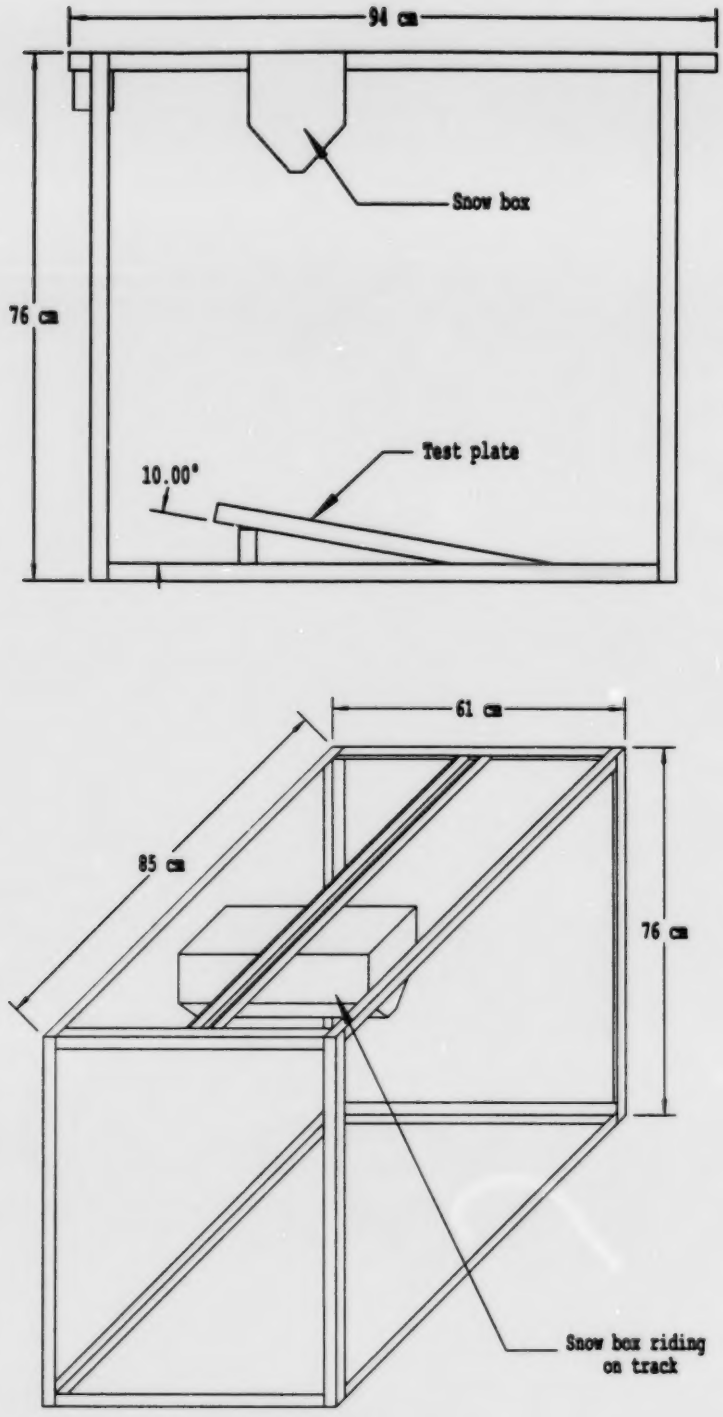


Figure 9: Sketches of the Snow Distribution System

4.3 Plate Set-up

For all tests except the frost tests, the candidate fluid was applied on 500 mm long x 300 mm wide x 3.2 mm thick polished aluminum alloy AMS 4037 plates placed on a 10° inclined set-up. The average roughness of the plates was between 0.1 and 0.2 μm. Examples of such a set-up are shown in Figures 10, 11, and 12. Each panel was surrounded with small 100 mm x 100 mm aluminum pans or plates for ice catch. The plates were 1.6 mm thick and were used for the freezing fog tests. The pans were 0.8 mm thick with an all-around rim 15 mm high and were used for the freezing drizzle, light freezing rain, and rain a on cold-soaked wing tests. For icing intensity calibration, twelve 100 mm x 100 mm aluminum plates or pans were placed on each large plate.

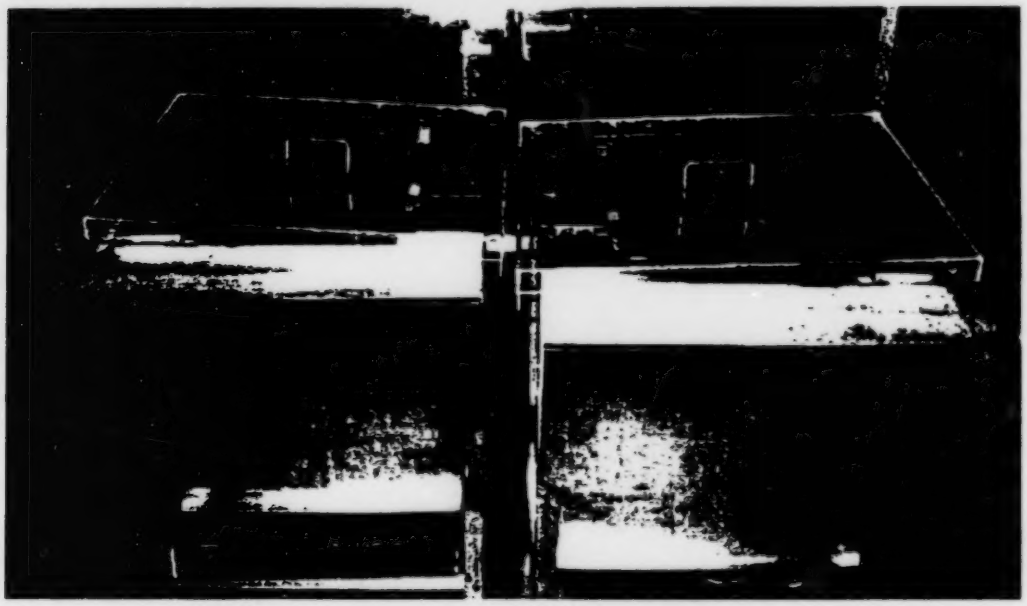


Figure 10: Example of Plate Support

Figure 11: Example of Plate Set-up

For the frost tests, the candidate fluid was applied on 300 mm long x 100 mm wide x 1.6 mm thick polished aluminum alloy AMS 4037 plates placed on the frosticator described in section 4.2.3. The average roughness of the plates was between 0.1 and 0.2 μm. Three panels for the fluid and nine 100 mm x 100 mm x 1.6 mm aluminum plates for the ice catch were positioned on the frosticator, as shown in Figures 13 and 14. For icing intensity calibration, three 100 mm x 100 mm aluminum plates or pans replaced each panel.

For the rain on a cold-soaked wing tests, a cold soak box filled with propylene glycol diluted 65/35 with water was placed underneath the panel (see Figure 15). The box was contained within a 25 mm polysterene insulating jacket (RSI = 1.3) during the test.

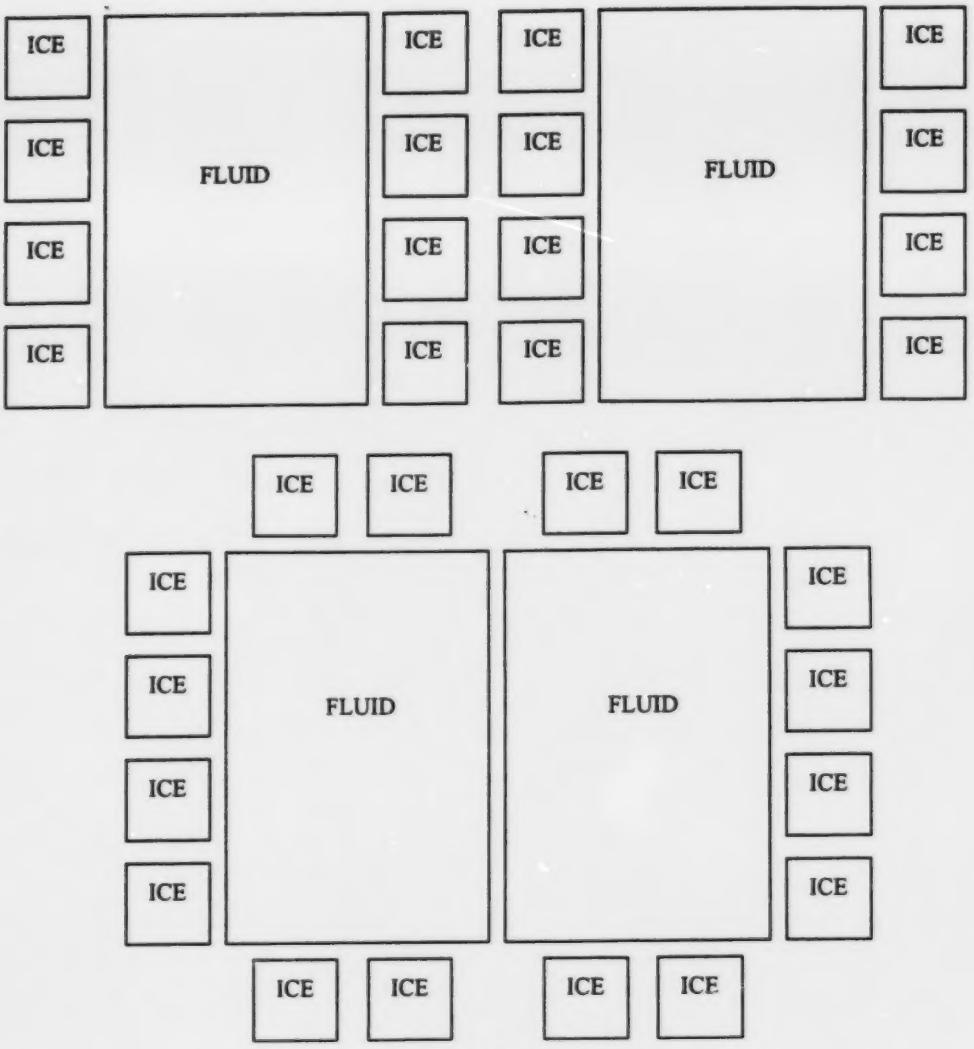


Figure 12: Sketch of Large Plate Set-ups

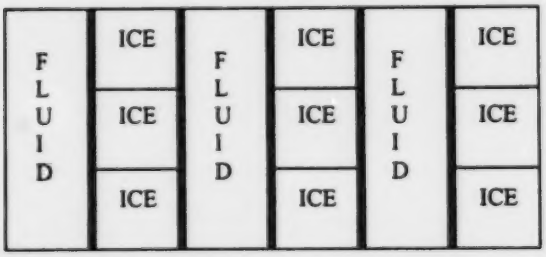


Figure 13: Sketch of the Frosticator Plate Set-up

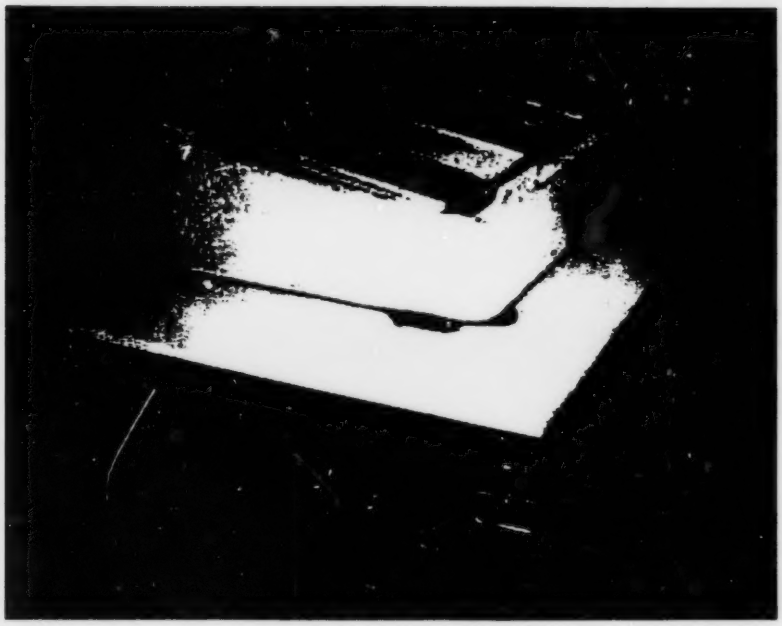


Figure 14: Frost Plate Set-up

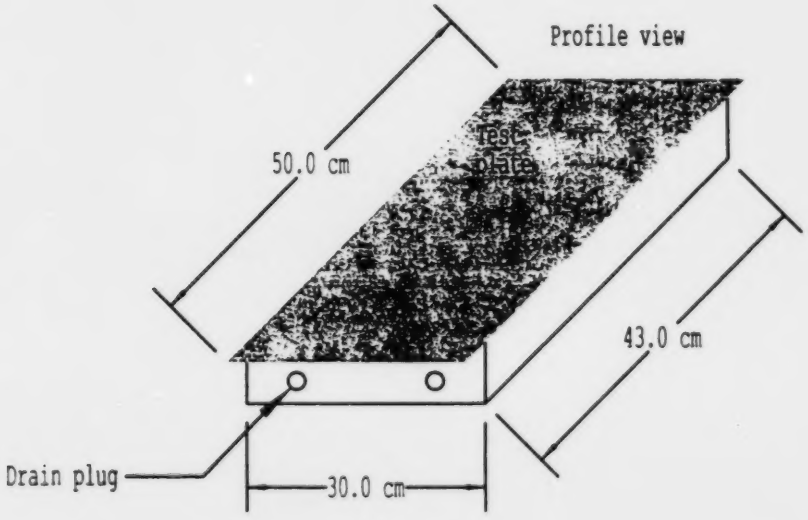


Figure 15: Sketch of the Cold Soak Box

4.4 Test Parameters

The validity of the tests depends essentially on three factors: the air temperature, the plate temperatures, and the icing intensity. Recordings of the air and plate temperature ensure that these parameters are maintained at their prescribed values during the test. Air temperature sensors, located within 1.5 m from the plates, and thermocouples placed underneath the plates were linked to a computer, which recorded the air and plate temperatures during the tests. The sampling rate on which the average and standard deviations were calculated is two data per second. The relative humidity was also recorded.

The horizontal component of the air velocities measured in the UQAC facilities 50 mm above the center line of the test plates is shown in Table 3 for each type of test as well as the limits proposed in the specification. The value measured at UQAC for the frost tests is higher than that of any other test, because frost tests require a room with a relatively small volume to achieve a high relative humidity level, but at the same time the heat exchangers must be powerful to maintain a very low temperature for a long period of time (up to 12 hours). Powerful heat exchangers in a small room create more draft than less powerful heat exchangers, or powerful heat exchangers used in a large room.

Table 3: Horizontal Air Velocity

PRECIPITATION TYPE	HORIZONTAL AIR VELOCITY (m/s)	
	UQAC	Specification
Frost	0.6	≤ 0.6
Freezing Fog	0.2	≤ 0.4
Snow	0.3	≤ 0.6
Freezing Drizzle	0.4	≤ 1.0
Light Freezing Rain	0.4	≤ 1.0
Rain on a Cold-Soaked Wing	0.4	≤ 1.0

5. CALIBRATION

Calibration tests were conducted for each type of precipitation to establish that even and reproducible ice formation occurred over the surface of the test plates, that is:

- the icing intensity targeted for the test was within an acceptable range; and
- the icing intensity over the surface of the panels exhibited a good distribution.

To carry out this evaluation, ice catch measurements were performed under each condition. To assess the ice catch, a total of at least twenty 100 mm x 100 mm ice catch plates or pans were used. Each 500 mm x 300 mm test panel, which would be coated with a fluid for a fluid test, was replaced with at least twelve 100 mm x 100 mm plates or pans during a calibration test (Figure 16). Pans were used when the precipitation needed to be contained, as is the case for the freezing drizzle, freezing rain, or rain on a cold-soaked wing tests, where the droplets do not freeze on impact and run down the slope. At -10°C, freezing drizzle droplets freeze on impact; therefore, calibration tests were performed at this temperature to compare the ice catch on the plates with that in the pans. They showed no significant difference between the ice catches. For the freezing fog and frost tests, plates were used since the ice is formed on contact with the plate. For convenience, only the term "plate" will be used in this section to refer to both plate and pan.

The preweighed plates were weighed upon completion of each test and the difference in the recorded weights was the ice catch for that plate. The average ice catch on the plates replacing the fluid test panel was calculated, as well as the average ice catch on the reference plates. It was the ratio between these two values that was used to estimate the icing intensity during a fluid test run, when only the reference plates were available, that is:

from calibration test:

$$Ratio = \frac{I_{plate}}{I_{ref}}$$

where:

Ratio is the ratio of the ice catch over the plate replacing the test plate with respect to the ice catch over the reference plates;

I_{plate} is the average ice catch on the plate replacing the test panel; and

I_{ref} is the average ice catch on the reference plates.

During an anti-icing endurance test run, the ice catch was measured on the reference plates and then multiplied by the ratio calculated above based on a calibration test performed under the same conditions. The resulting value was the estimated icing intensity over the test panel:

for fluid test run: $Estimated\ I_{plate} = Ratio \times I_{ref}$

where I_{ref} was measured during a fluid test and the ratio was determined in a previous calibration test.

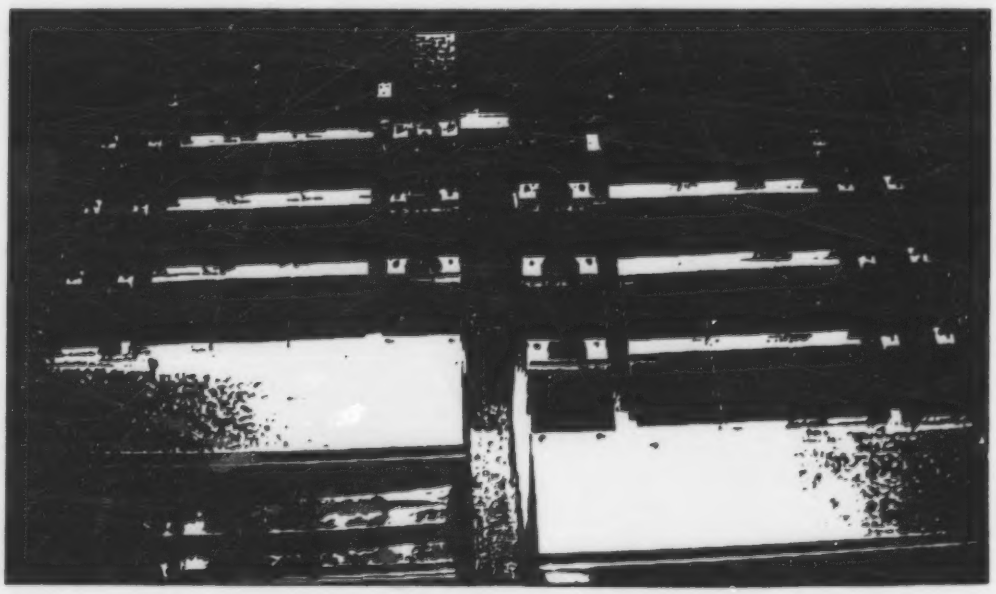


Figure 16: Example of a Calibration Set-up

6. TOLERANCE ON TEST PARAMETERS

A tolerance on each test parameter must be defined to ensure that all tests are performed in the same way in different facilities, at different times, by different people, while achieving good test reproducibility. The foregoing tolerances are based on Annex A of AMS1428C [1], which describes anti-icing performance tests.

6.1 Tolerance on Equipment and Instrumentation

- Plate finish: average roughness between 0.1 and 0.2 μm
- Plate slope: 10° ± 0.2
- Temperature sensor accuracy: 0.5°C

6.2 Tolerance on Icing Intensity

For the Water Spray Endurance Test defined in AMS1428C, which has a target icing intensity of 5 g/dm²/h, the tolerance is defined by the maximum variation allowed about the target intensity, which is 4 percent. For the High Humidity Endurance Test defined in the same specification, the target icing intensity is 0.3 g/dm²/h and the maximum variation allowed about the target is 17 percent. It is indeed more difficult to maintain an even distribution when the intensity is lower. Very strict tolerances would have been difficult to achieve on a large test area, therefore the tolerances have been relaxed, as described below.

6.2.1 Tolerance on the Average Icing Intensity

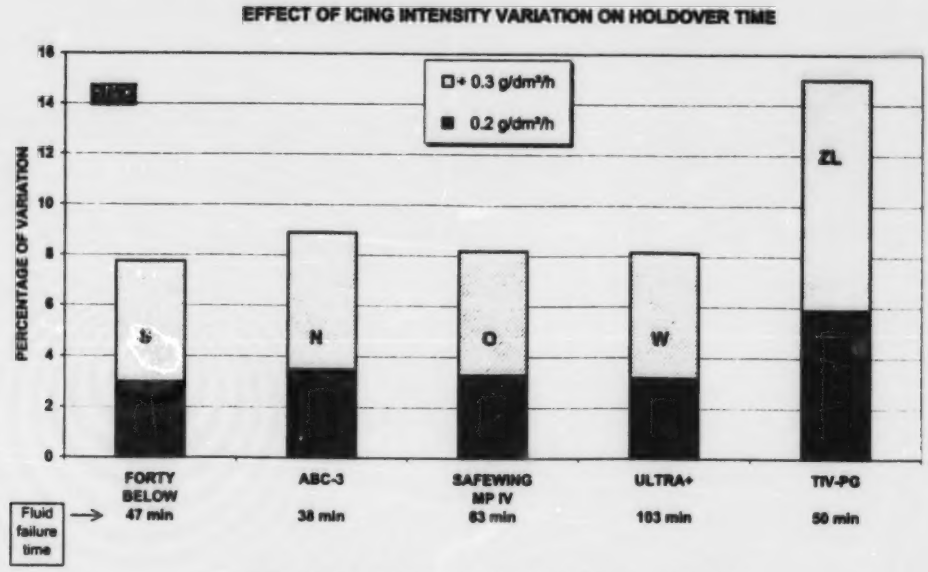
The average icing intensity measured during the calibration tests and estimated during the fluid tests must fall within the following ranges (generally 4 percent of the target intensity):

- 75.0 ± 1.5 g/dm²/h (rain on a cold-soaked wing)
- 25.0 ± 1.0 g/dm²/h (light freezing rain, snow, and rain on a cold-soaked wing)
- 13.0 ± 0.5 g/dm²/h (freezing drizzle and light freezing rain)

- 10.0 ± 0.4 g/dm²/h (snow)
- 5.0 ± 0.2 g/dm²/h (snow, freezing fog, and freezing drizzle)
- 2.0 ± 0.2 g/dm²/h (freezing fog)

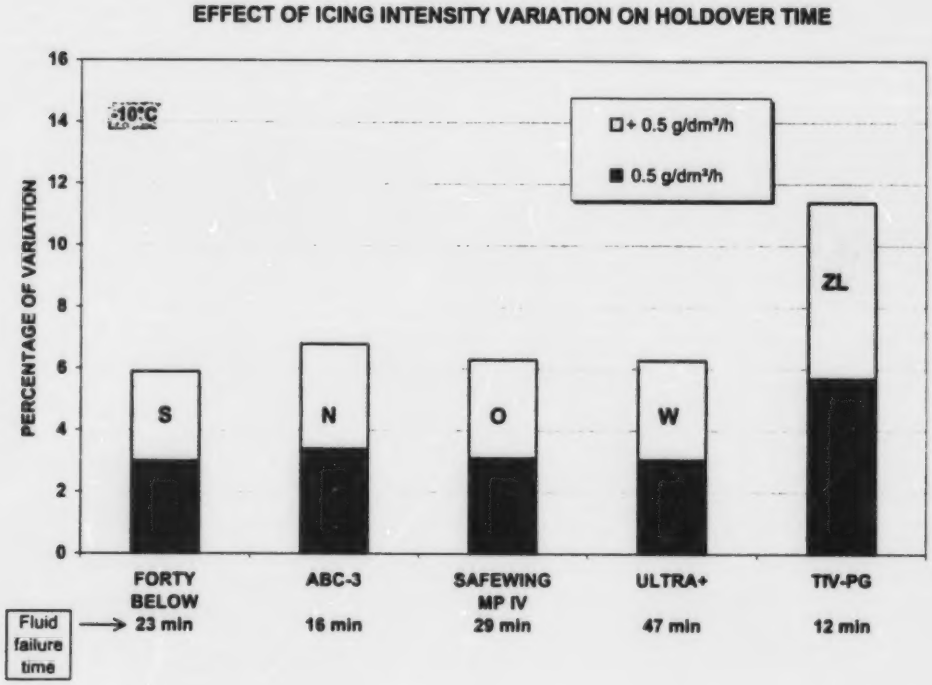
  For the low intensity of freezing fog, 2 g/dm²/h, the variation allowed has been increased to 10%.
- 0.20 ± 0.02 g/dm²/h (frost at 0 and -3°C)
- 0.13 ± 0.01 g/dm²/h (frost at -14°C)
- 0.05 ± 0.01 g/dm²/h (frost at -25°C)

Figures 17 to 19 show how the holdover time can be affected by the variation of the icing intensity for various target intensities. These figures are based on snow test data presented in a previous report entitled *Development of Laboratory Test Procedures to Replace Field Anti-icing Fluid Tests,* prepared for the Transportation Development Centre [2]. A regression analysis on these data permitted the determination of power laws to calculate the holdover time as a function of icing intensity for various fluids. This function was in turn used to compute the values shown in Figures 17, 18 and 19. The shaded columns show the percentage of variation of the holdover time when the tolerance on the average icing intensity is 4 percent. The solid columns show how an increase of the tolerance, by 6 percent in the case of Figure 17 and 4 percent in the case of Figure 18, would affect the holdover times. These figures show that the holdover time variation fluctuates between 3 and 6 percent when the tolerance is 4 percent, which is quite acceptable, but increases to between 6 and 14 percent when a further tolerance between 4 and 6 percent is added, which is too high.



TIV-PG is a propylene glycol-based Type IV fluid

Figure 17: Effect of Variation of the Average Icing Intensity on Holdover Time (5 g/dm²/h)



TIV-PG is a propylene glycol-based Type IV fluid

Figure 18: Effect of Variation of the Average Icing Intensity on Holdover Time (13 g/dm²/h)

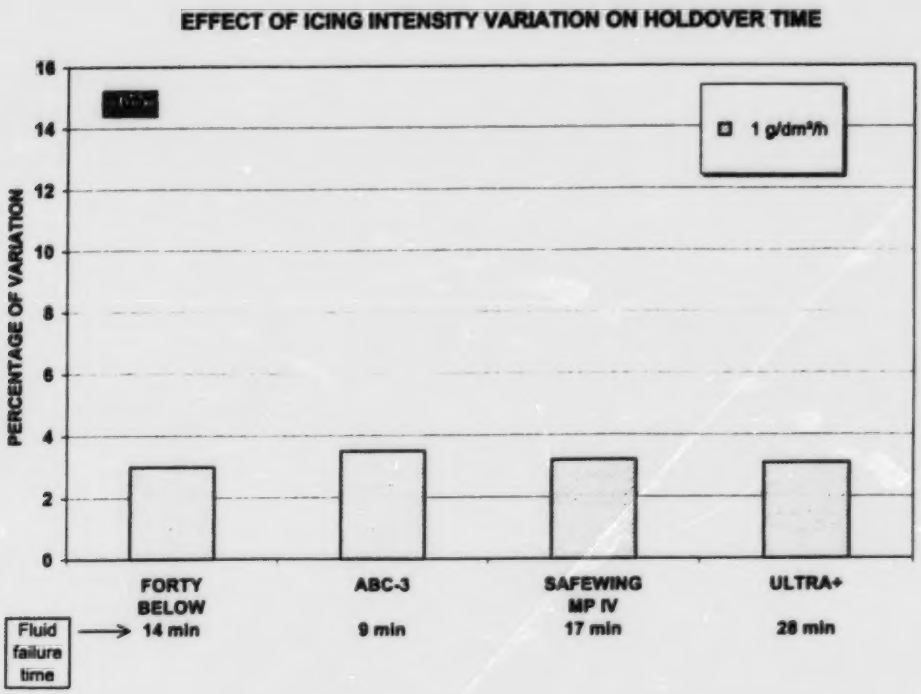


Figure 19: Effect of Variation of the Average Icing Intensity on Holdover Time (25 g/dm²/h)

6.2.2 Tolerance on Icing Intensity Distribution

The icing intensity distribution over the test panel was evaluated during the calibration tests by the standard deviation calculated over the ice catch measured on each 100 mm x 100 mm plate or pan; this meant at least twelve plates or pans per test panel for the freezing fog, freezing drizzle, light freezing rain, rain on a cold-soaked wing, and snow calibrations, and eighteen plates for the frost calibrations. The standard deviation must fall within 6 percent of the average intensity, except for frost where the tolerance was increased as follows:

- 0.03 g/dm²/h for a target intensity of 0.20 g/dm²/h
- 0.01 g/dm²/h for a target intensity of 0.13 g/dm²/h
- 0.01 g/dm²/h for a target intensity of 0.05 g/dm²/h

6.3 Tolerance on Air Temperature

For the Water Spray Endurance Test and the High Humidity Endurance Test defined in AMS1428C, the variation allowed about the target air temperature of 5°C is 0.5°C. The same tolerance was used here in most cases.

6.3.1 Tolerance on Average Air Temperature

The average air temperature measured during a calibration test and a fluid test must fall within 0.5°C for any target temperature except in the case of the frost tests. Because of technical limitations, the tolerance on air temperature in the frost case was set as follows:

- 0.5°C at 0, -3 and -14°C
- 2.0°C at -25°C and below -25°C

Figures 20 and 21 show how the holdover time can be affected by the variation of the air temperature for two target temperatures. These figures are based on snow test data presented in a previous report [2]. A curve fitting technique on these data permitted the interpolation of the holdover time as a function of air temperature for various fluids. The values shown in Figures 20 and 21 are the results of these interpolations. The shaded columns show the percentage of variation of the holdover time when the tolerance on the average air temperature is 0.5°C. The solid columns show how an increase of the tolerance by an additional 0.5°C would affect the holdover times. These figures show that the holdover time variation fluctuates between 0.5 and 6 percent when the tolerance is 0.5°C, which is quite acceptable, but increases to between 1 and 11 percent when a further 0.5°C tolerance is added, which is too high.

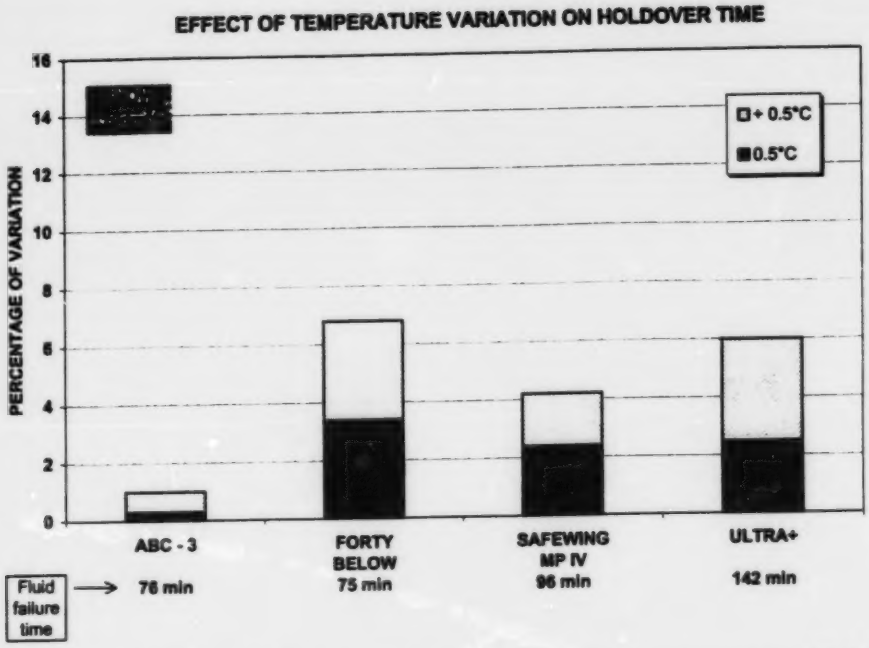


Figure 20: Effect of Variation of the Average Air Temperature on Holdover Time (-4°C)

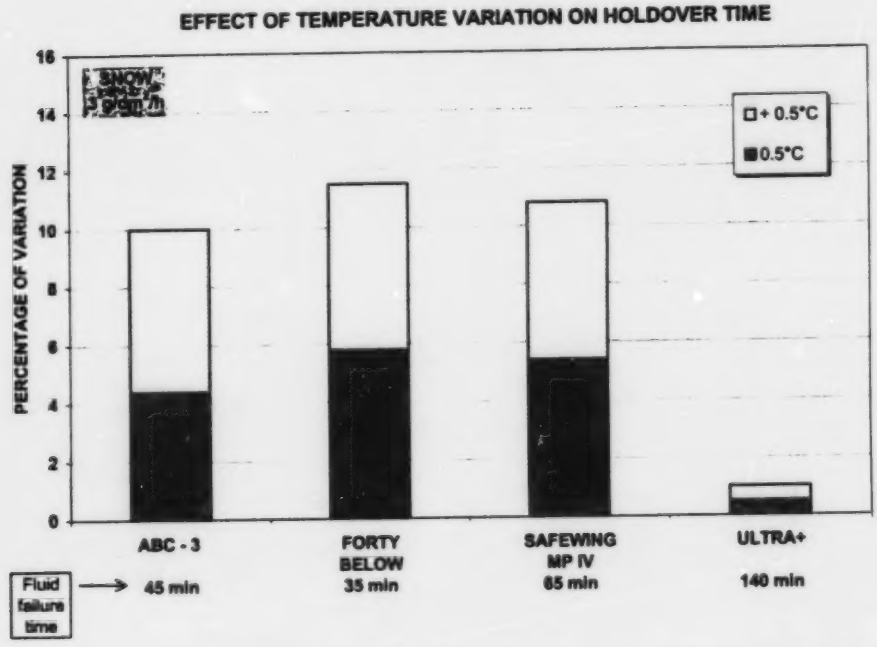


Figure 21: Effect of Variation of the Average Icing Intensity on Holdover Time (-14°C)

6.3.2 Tolerance on Air Temperature Variation

The variation of the air temperature during a test was evaluated by the standard deviation calculated on data collected during the tests with a sampling rate of at least one datum per minute. In UQAC chambers the sampling rate is two data per second. The standard deviation must fall within the following limits:

- 0.3°C at all temperatures for freezing drizzle, light freezing rain, and rain on a cold-soaked wing tests
- 0.2°C at -3 and -14°C for snow tests
- 0.5°C at -25°C and below -25°C for snow tests
- 0.2°C at 0, -3 and -14° C for frost tests
- 0.5°C at -25°C and below -25°C for frost tests

These limits were based on UQAC and NRC data.

7. TESTS

7.1 Methodology

The test procedure was based on the certification procedure described in AMS1428C Annex A. This procedure was adjusted for each test according to the descriptions presented in the previous sections. Furthermore, other modifications were made according to the November 1997 meeting. For example, the quantity of fluid applied to the 500 mm x 300 mm plate was 500 mL; the Type IV fluids were applied at test temperature and the Type I fluids at 20 ± 5°C; the fluids were sheared as specified in AMS1428C except for the delay between the shearing and the application of the Type IV fluids, which was increased to more than two hours because of the need for fluid refrigeration; and finally, the fluids were applied under precipitation.

Over four hundred tests, including calibration and fluid tests, were conducted under the various types of precipitation. The calibration tests assessed whether the tolerances on the test parameters defined in the previous section could be met easily enough for the procedure to be functional. The fluid tests verified the effect of the test parameter tolerance on the holdover times, and identified problems and lack of information in the test procedures.

7.2 Freezing Fog Tests

Some of the freezing fog test data are presented in Figures 22, 23, and 24. Under certain conditions, the Type IV fluids seemed to exhibit too much variation. The operator noticed that the fluid flow, at the time of pouring, varied from test to test. This problem will be examined more closely in section 7.8. The only other notable observation from these tests is that the failure can easily be missed at -25°C. At this low temperature, the fluid is dull and the ice contamination consists of tiny (about 25 μm), white frozen water droplets, which are very difficult to distinguish from the fluid itself.

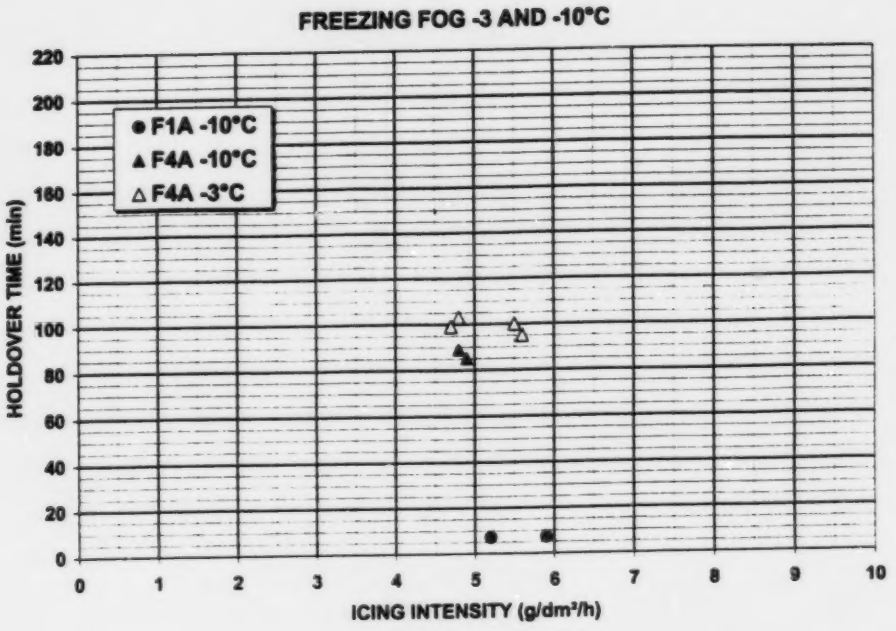


Figure 22: Freezing Fog Tests at -3 and -10°C

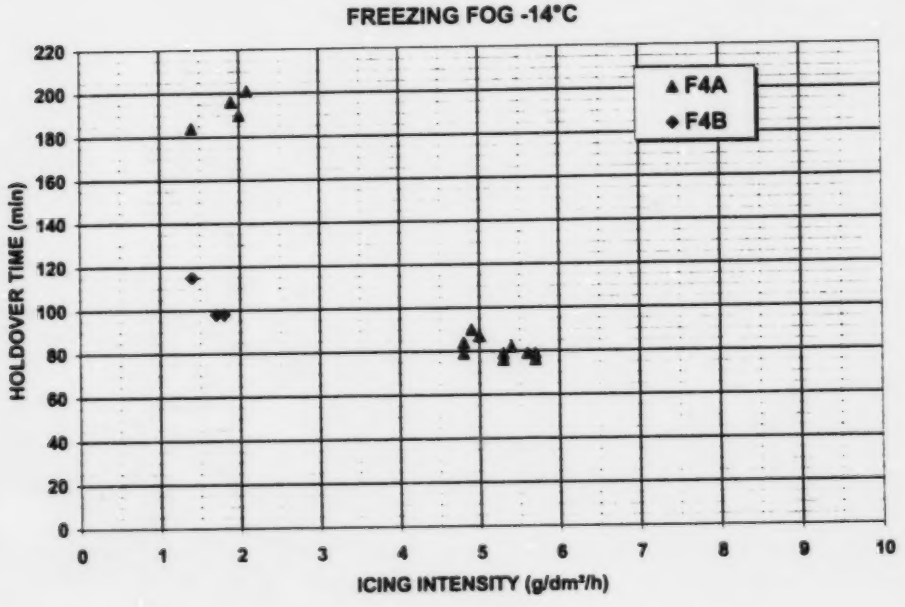


Figure 23: Freezing Fog Tests at -14°C

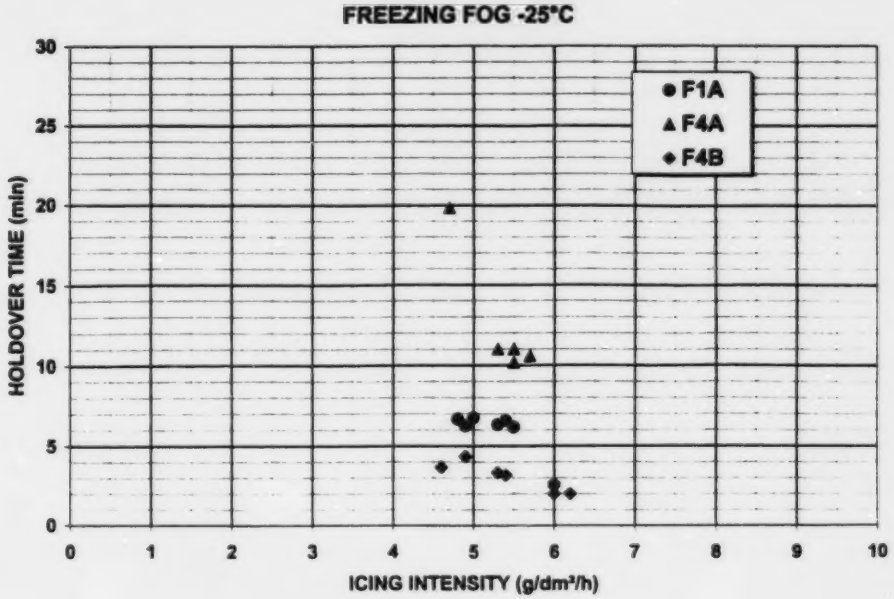


Figure 24: Freezing Fog Tests at -25°C

7.3 Freezing Drizzle Tests

Some of the freezing drizzle test data are presented in Figures 25 and 26. Under certain conditions, the Type IV fluids seem to exhibit too much variation, especially at -10°C at the 5 g/dm²/h icing intensity. This will be examined more closely in section 7.8. Figure 27 presents the Type I fluid data.

The failure mode for freezing drizzle tests is usually an ice front or a slush formation.

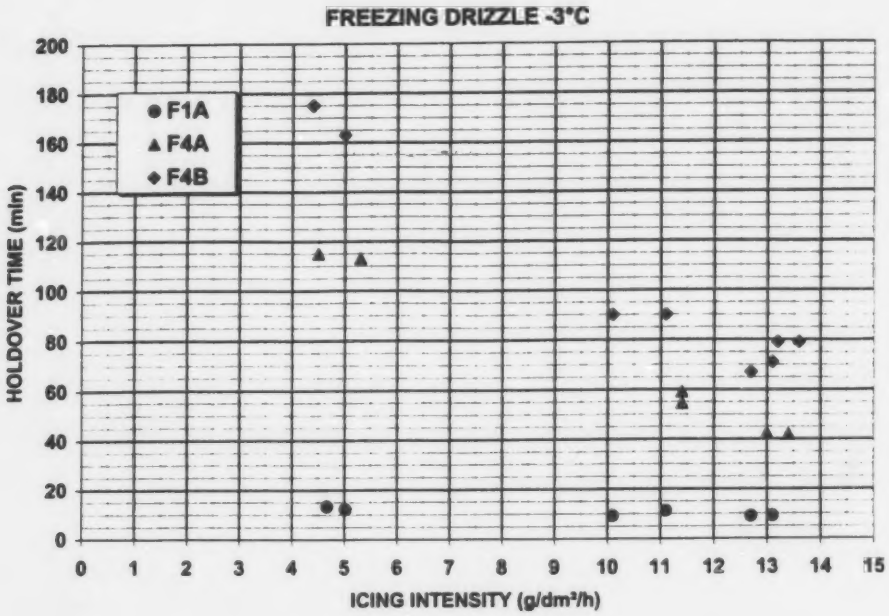


Figure 25: Freezing Drizzle Tests at -3°C

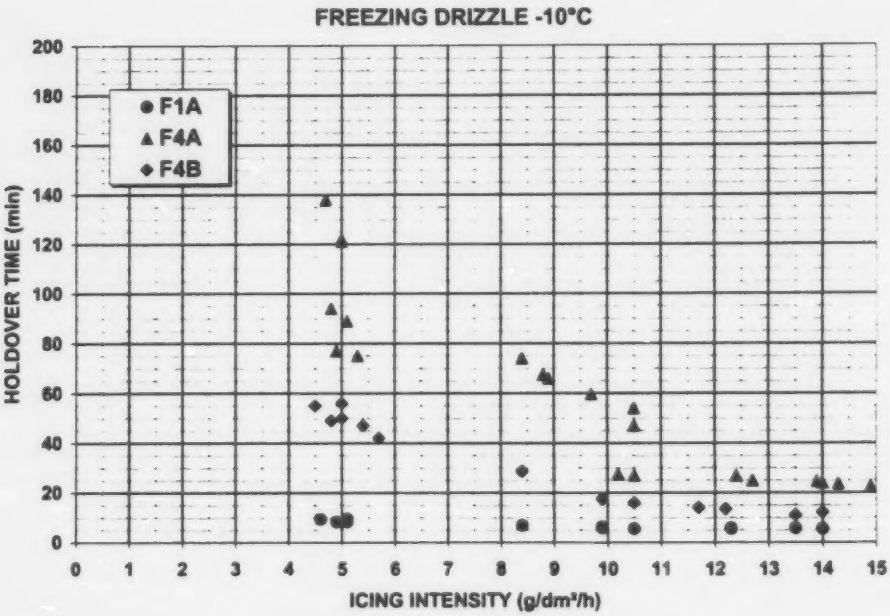


Figure 26: Freezing Drizzle Tests at -10°C

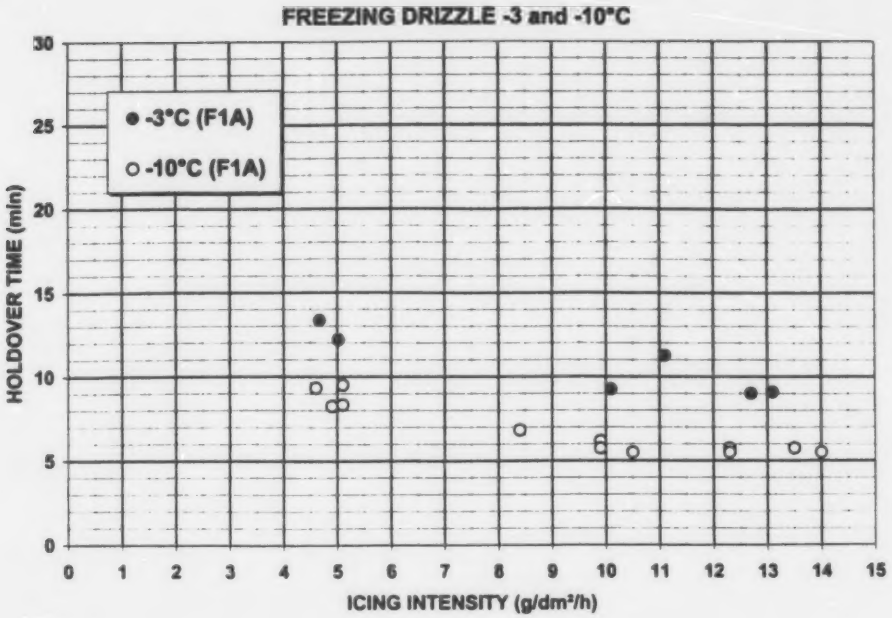


Figure 27: Type I Freezing Drizzle Tests at -3 and -10°C

7.4 Light Freezing Rain Tests

Some of the light freezing rain test data are presented in Figures 28 and 29. Under certain conditions, the Type IV fluids seem to exhibit too much variation, especially at -10°C at the 13 g/dm²/h icing intensity. (See section 7.8 for further details). Examples of ice formation at -3°C are given in Figures 30 and 31. At -10°C under a 25 g/dm²/h icing intensity, most fluids fail by contamination with clear pieces of ice, which are very difficult to see.

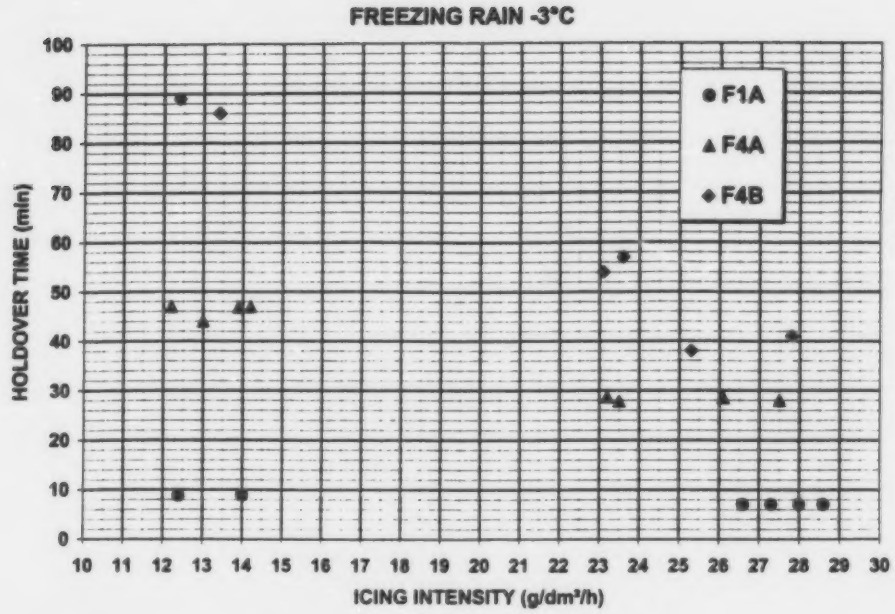


Figure 28: Light Freezing Rain Tests at -3°C

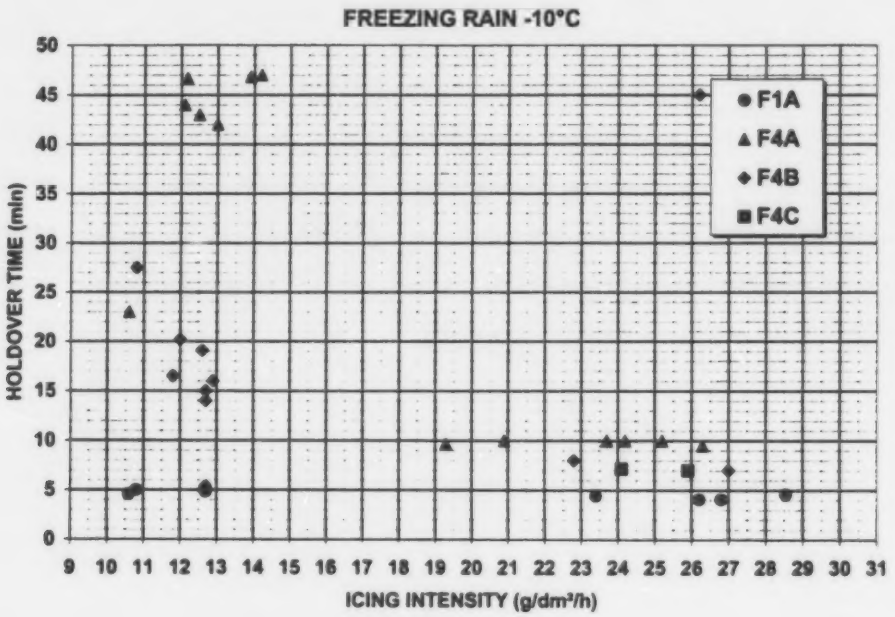


Figure 29: Light Freezing Rain Tests at -10°C

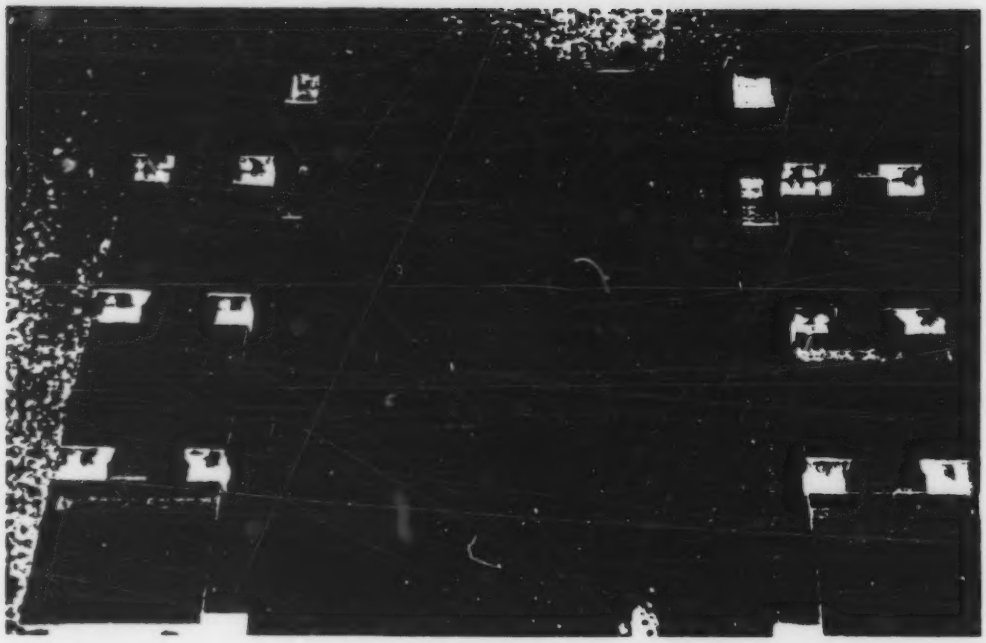


Figure 30: Ice Formation on Type I Fluid

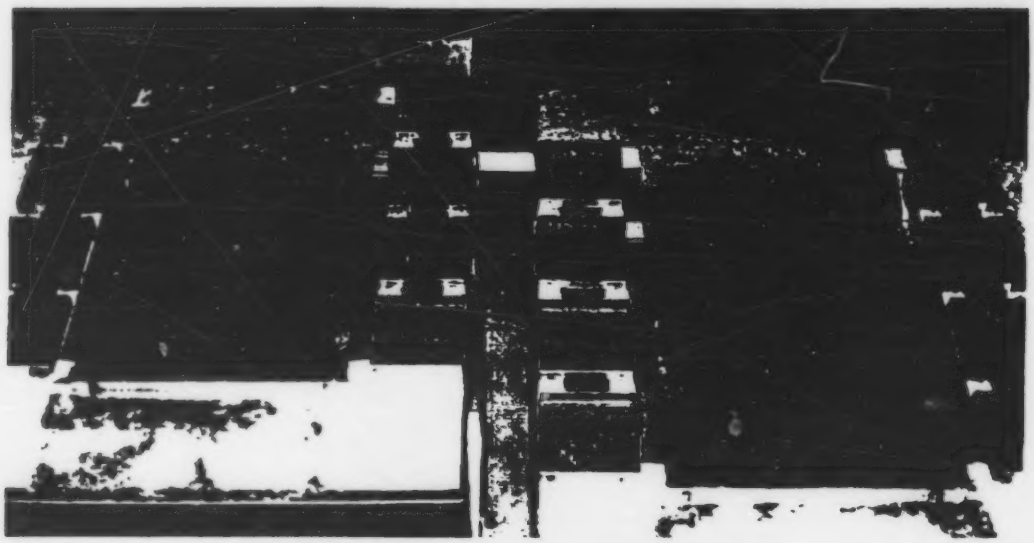


Figure 31: Ice Formation on Type IV Fluid

7.5 Frost Tests

Several calibration tests were performed to establish feasible testing conditions. Producing and maintaining a high relative humidity level at low temperatures for extended periods of time proved to be quite difficult. The main problem is that frost is immediately collected on surfaces where the temperature is lower than that of the humid air. This characteristic is used for the fluid test since the test panels are refrigerated at a temperature 3°C below the ambient air temperature. However, the main element in the chamber colder than the ambient air is the heat exchanger. Therefore, over a period of time, frost tends to accumulate on the heat exchanger and decrease its efficiency. At very low temperatures, it becomes difficult to maintain a high relative humidity level, because the humidity deposits on the cold room's heat exchanger as much as on the test panels. The testing conditions that could be achieved several times are presented in section 4.4. A few tests were performed with Type IV fluids. The results are presented in Table 4.

Table 4: Frost Test Data

Fluid Code	Air Temperature (° C)	Fluid Temperature (° C)	Icing Intensity (g/dm²/h)	Relative Humidity (%)	Endurance Time
F4A	-25.0	-27.9	0.06	71.3	> 12 hours
F4B	-25.0	-27.9	0.06	71.3	4 h 50 min
F4C	-25.0	-27.9	0.06	71.3	2 h 10 min
F4A 75/25	-3.0	-5.9	0.21	98.1	4 h 30 min
F4B 75/25	-3.0	-5.9	0.21	98.1	> 5 hours
F4C 75/25	-3.0	-5.9	0.21	98.1	> 5 hours
F4A 75/25	-14.0	-17.4	0.13	87.9	> 5 hours
F4B 75/25	-14.0	-17.4	0.13	87.9	> 5 hours
F4C 75/25	-14.0	-17.4	0.13	87.9	> 5 hours
F4A 50/50	-3.0	-6.0	0.19	98.6	1 h 30 min
F4B 50/50	-3.0	-6.0	0.19	98.6	> 3 hours
F4B 50/50	-3.0	-6.0	0.19	98.6	> 3 hours

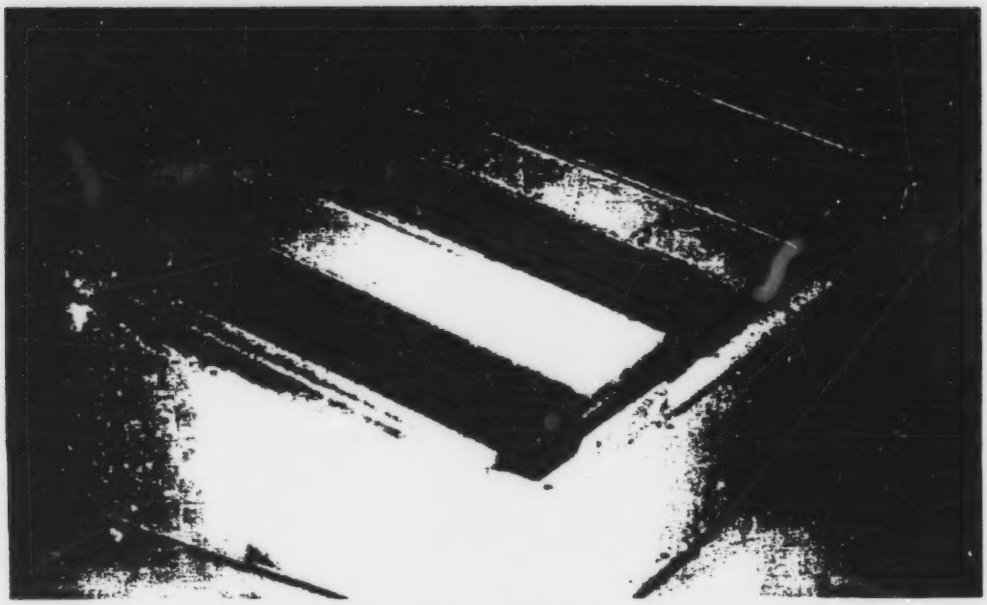


Figure 32: Example of a Frost Test

7.6 Rain on a Cold-Soaked Wing Tests

Several tests were performed to become familiarized with the procedure developed by APS, which is described in the report entitled *Validation of Methodology for Simulating a Cold-Soaked Wing* prepared for the Transportation Development Centre [3]. To facilitate the cleaning process, AMIL used a removable panel as the test panel rather than the upper surface of the cold soak box. The two graphs, presented in Figures 33 and 34, show that the presence of a removable panel does not affect the temperature evolution. The graphs present the temperature inside the box, T_{box}, and the temperature on the surface of the panel, $T_{p,\,center}$, as functions of time. Figure 33 corresponds to the case where the panel is removable, and Figure 34 to the case where the upper surface of the cold soak box is the test panel. A superimposition of the two graphs shows that the curve of the plate temperature is the same in both cases. This shows that the use of a removable test panel does not affect the test as described in the APS report [3].

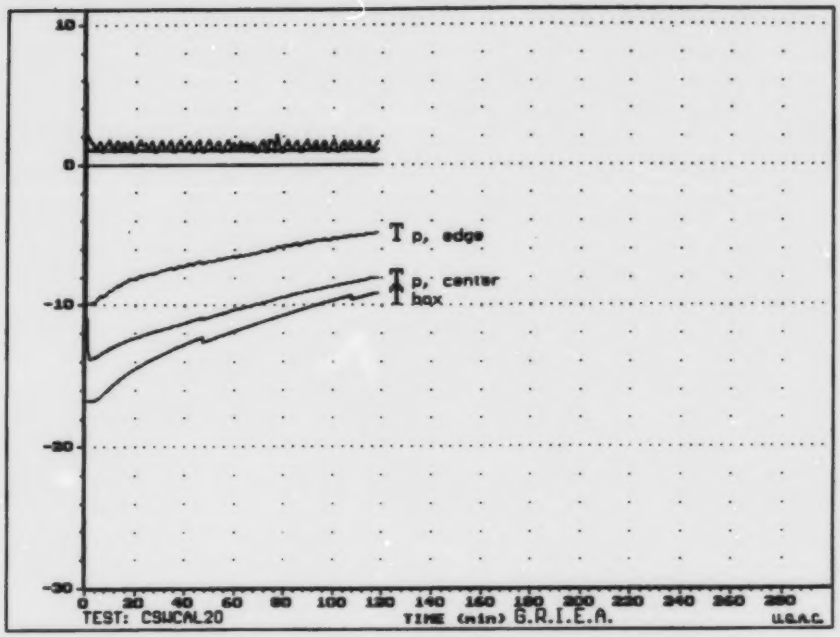


Figure 33: Surface Temperature of the Cold Soak Box Equipped with a Removable Panel

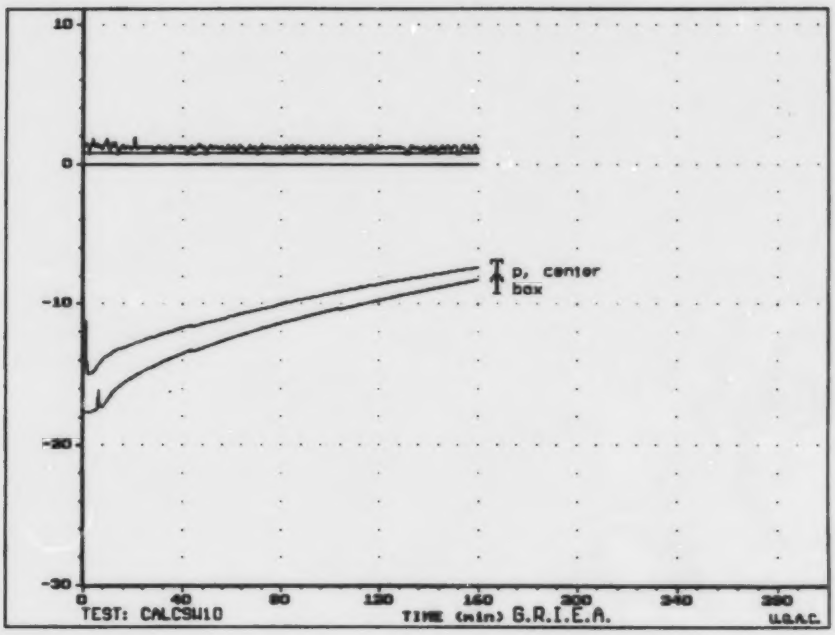


Figure 34: Surface Temperature of the Cold Soak Box without Removable Panel

7.7 Snow Tests

7.7.1 Initial Data on Laboratory Indoor Snow

The basic data pertaining to the Transport Canada report on snow simulation [2] and comparisons between outdoor and indoor snow tests are presented in Figures 35 to 38. The density of the natural snow collected outdoors on the ground and used in the laboratory was relatively low (0.15 g/cm³). The snow was distributed manually over the test panel. These data were presented in November 1997, at the meeting of the Workgroup on Laboratory Methods to Derive Holdover Time Guidelines held in Montreal [4].

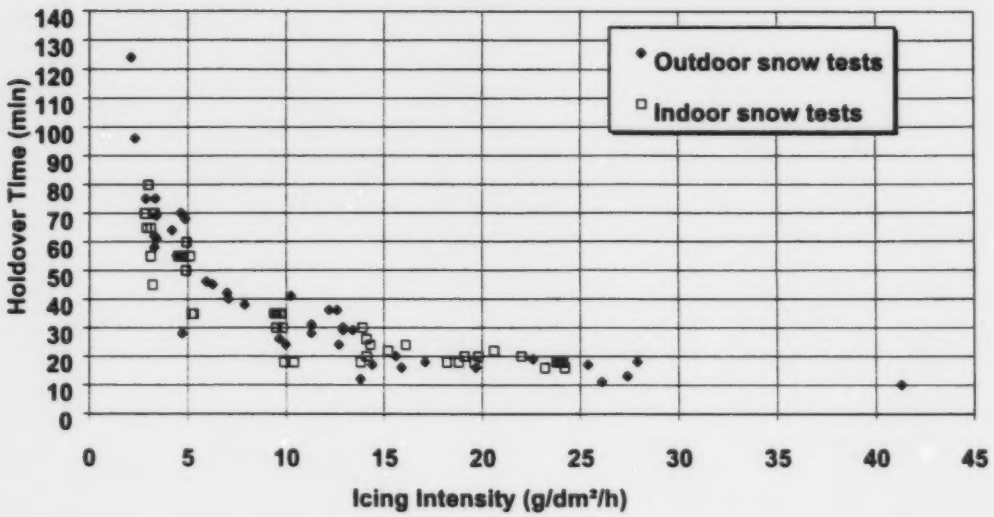


Figure 35: Forty Below Initial Snow Data

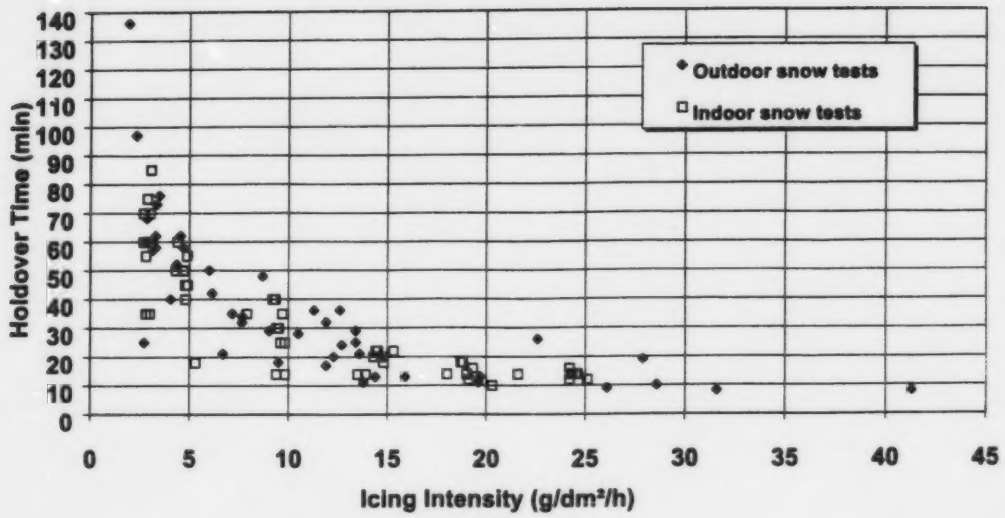


Figure 36: ABC-3 Initial Snow Data

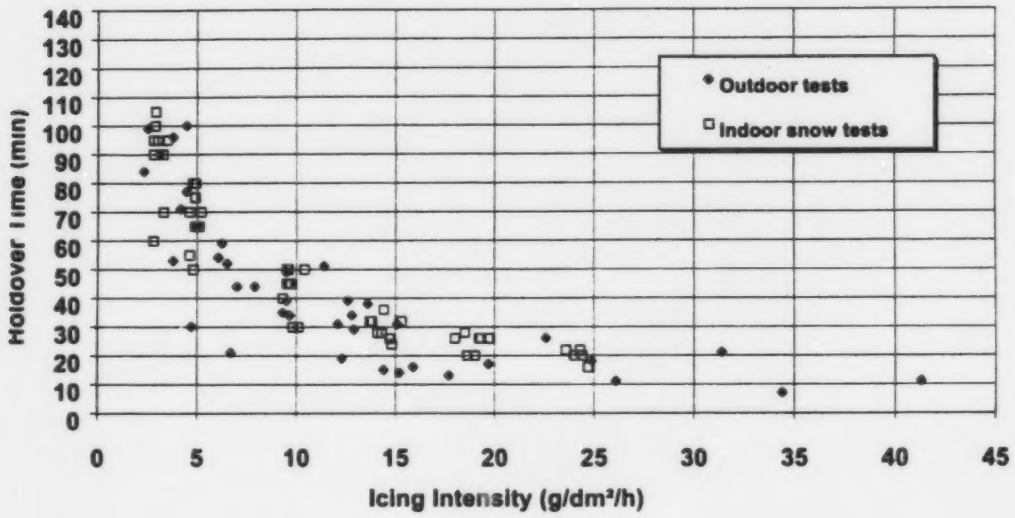


Figure 37: Safewing MP IV 1957 (1st Generation) Initial Snow Data

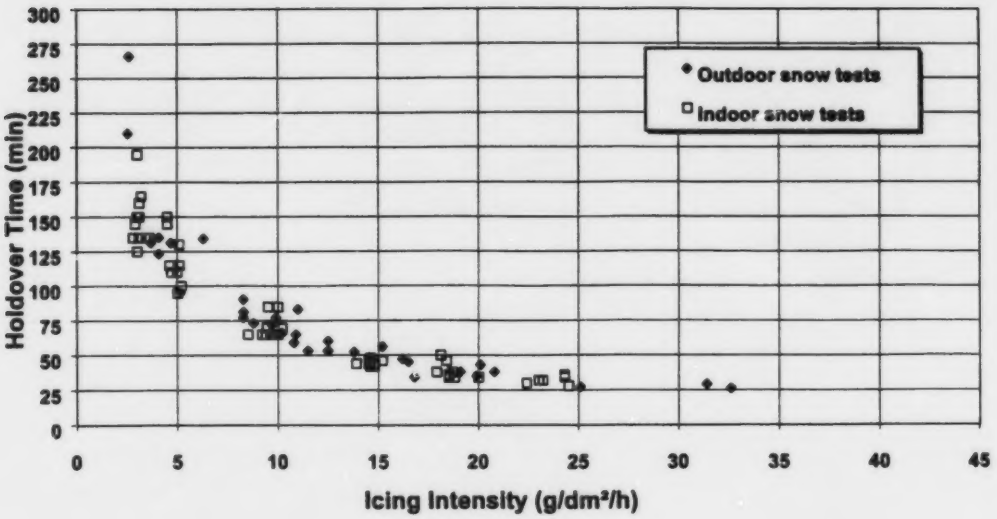


Figure 38: Ultra+ Initial Snow Data

As can be seen in Figures 35 to 38, the indoor data points agree well with the outdoor data points for all fluids except for Safewing MP IV 1957 (1st generation). For that fluid, several outdoor data points are below those of the indoor tests. These points correspond mostly to cases of snow pellet precipitation as recorded during the test runs, although at least one corresponds to a snowflake event, which had particularly large flakes. This fluid did not readily absorb snow particles when they brought more water into a small area of the fluid, which usually occurs with small or normal sized snowflakes. This could happen as well in the case of very wet snow, although the overall intensity may be well within the range considered for the holdover times.

7.7.2 Improvement on Laboratory Indoor Snow

As written in the minutes of the meeting (Note (a) on page 9 in [4]), an AMIL representative suggested running "tests with larger sized artificial snow (made from larger droplets…)" to identify fluids with such behaviour, and in turn, an NCAR representative suggested using snow presenting a higher density. This

suggestion was then supported by AMIL, since the use of larger droplets in the snowmaking process automatically increases the snow density.

In the May 1998 SAE G-12 meeting in Vienna, AMIL presented data showing that it was indeed possible to produce, as outdoors, shorter times for Safewing MP IV, when using snow with a density of 0.25 g/dm³. It was also demonstrated that, as outdoors, the 0.25 g/dm³ density was not shortening Ultra+ performance. These data were obtained with a manual system. Section 7.7.3 provides data produced with the automated system described in section 4.2.4.

7.7.3 Automated Snow System Data

Snow test data obtained with the automated system described in section 4.2.4 are presented in Figures 39 and 40. They pertain to four different Type IV fluids, one ethelyne-glycol based and three propylene-glycol based. Table 5 and Figure 39 show data obtained when the ethylene-glycol based product, UCAR Ultra+, was tested at -10°C under two conditions. The first condition represents a low density snow, about 0.1 g/cm³, manually distributed with a sifter above the test panel every two or five minutes. The second condition represents a high density snow, about 0.25 g/cm³, distributed over the test panel by the automated system described in section 4.2.4.

Table 5: ULTRA+ Snow Data at -10°C

Icing Intensity (g/dm²/h)	Low Density Snow (manual) (min)	High Density Snow (automated) (min)
5	65	72
5	60	-
10	40	44
10	40	-
15	28	30
15	26	28
24	18	20
25	20	22
30	-	17
40	12	-
40	12	-

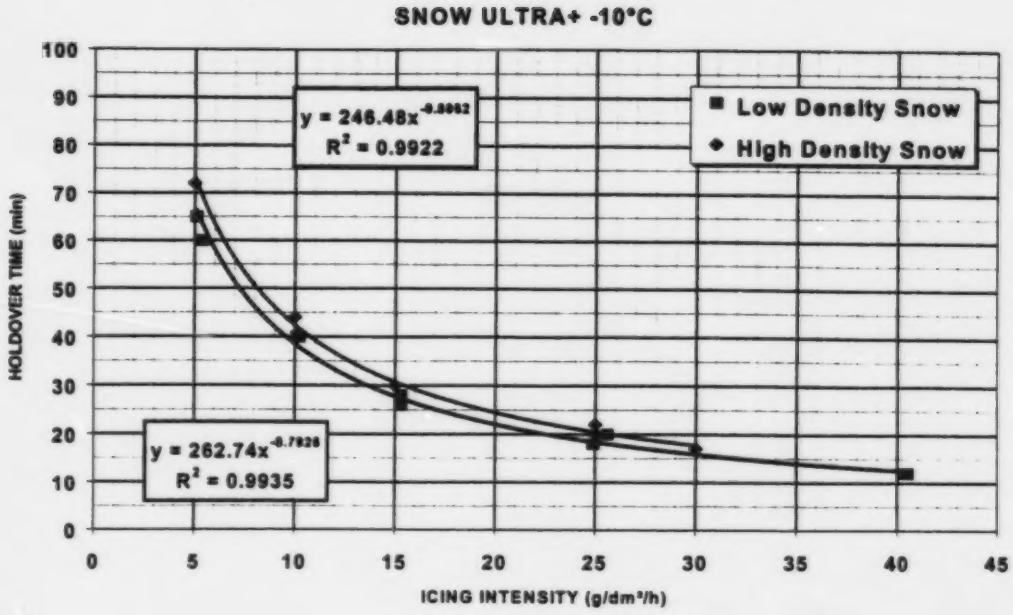


Figure 39: Snow Tests with UCAR Ultra+ at -10°C

Table 5 and Figure 39 show that the Ultra+ data obtained under the two conditions are very similar, although the high density snow endurance times are consistently a little longer than the low density snow endurance times. At this time, no conclusive explanation can be offered, especially since this phenomenon did not occur with the other fluids tested, as shown in the following discussion.

Figures 40 and 41 respectively show data obtained with three Type IV-PG fluids and two Type I fluids tested at -10°C under the two conditions previously described. As can be seen in Table 6, which summarizes all data, only Safewing MP IV 1957 (1st generation), which is the same fluid tested in the initial outdoor tests, presents a significant difference between the anti-icing endurance times recorded under the low and high snow density precipitation. All other fluids yield similar results under both conditions of snow density.

Table 6: Type IV Snow Data

Fluid Code	Temperature (°C)	Icing Intensity (g/dm²/h)	Low Density Endurance Time (min)	High Density Endurance Time (min)
Safewing MP IV 1957 (1st generation)	-10	10	26	15
Safewing MP IV 1957 (1st generation)	-10	25	12	8
F4B	-14	10	20	20
F4B	-14	25	10	10
F4C	-14	10	16	15
F4C	-14	25	6	8
ULTRA+	-10	10	40	44
ULTRA+	-10	25	20	20

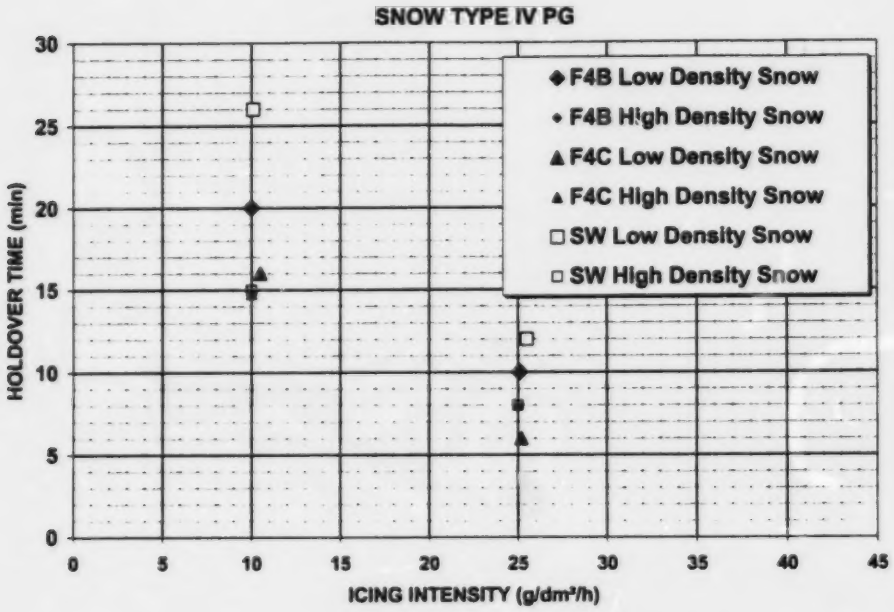


Figure 40: PG-based Type IV Snow Tests

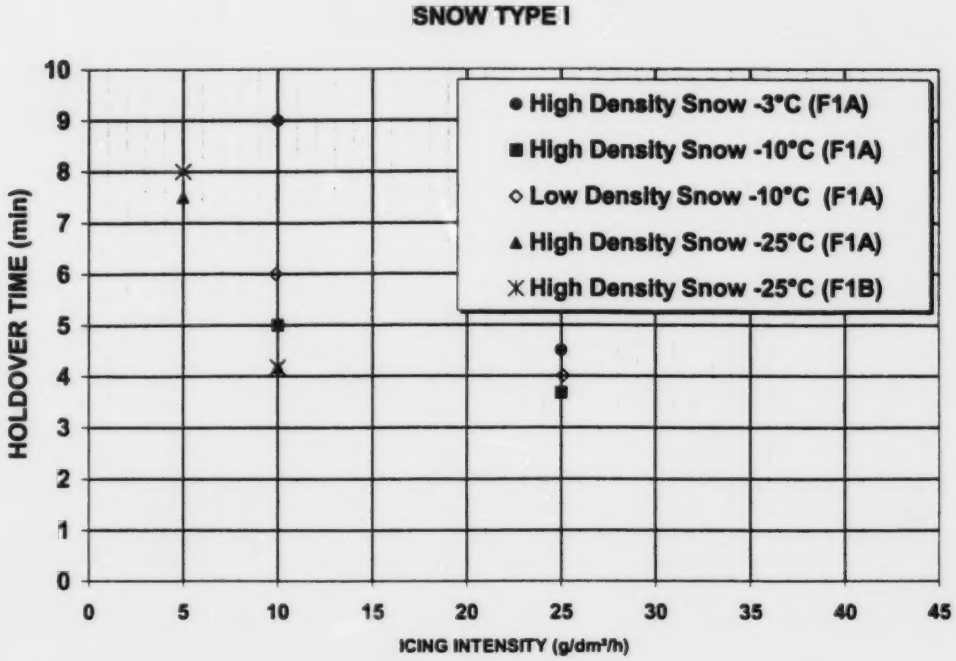


Figure 41: Type I Snow Tests

7.7.4 Role of Snow Density

The question of laboratory snow density should be studied in relation to fluid failure. Therefore, it is the density of the individual snowflake as it reaches the fluid surfaces which matters. A true snowflake is characterized by a lot of air space within its volume. AMIL artificial snow is made of an agglomerate of many tiny ice beads separated by a lot of air spaces, which thus provides a dissolving process quite similar to that of true snow. On the other hand, individual snowflakes constituted of large pure ice crystals have a much higher density, close to 0.9 g/cm³, and the dissolution process is therefore quite different from that of true snowflakes. However, this high density of the individual snowflakes may not register in a measurement of the snow density: if the ice flakes are collected in a container, they will pile up, trapping air between the flakes and the result is an apparent density much lower than that of the individual flakes. An indication of the problems created by a system producing ice flakes can be found in section 3.2 (pp. 11-13) of the FAA/AR-98/74 report entitled *Development of a Method to Test Holdover Times of*

Deicing and Anti-icing Fluids in a Cold Room Using Artificially Generated Snow [5], which shows that the data obtained with the system used in the laboratory were significantly shorter than the outdoor data. Indeed, the NCAR system uses a technique of shaving an ice core made of frozen water. The density of frozen water is usually close to 0.9. Therefore, any single piece of the ice cylinder will have a density between 0.8 and 0.9, no matter how small or how thin it is. Consequently, any "ice flake" reaching a tested fluid will have a density between 0.8 and 0.9, which is the probable cause of the observed shorter holdover times.

7.8 Holdover Time Variation

As shown in several previous sections, the holdover time variation was sometimes quite substantial although the tolerance on the test parameters had been respected. Test operators observed that the fluid flow on the test panels varied from test to test and suggested that the anti-icing endurance time variation could have resulted from variations in the fluid condition at the time of application. When poured, the fluid appeared more or less viscous with a texture more or less uniform. Therefore, tests were performed with several fluids under various intensities of shaking. To vary the shaking intensity, the sample was handled in several ways: no disturbance, repeated inversion of a bottle containing the fluid, and fairly vigorous shaking of the bottle. A few tests were performed with the fluids applied warm, at 20 ± 5°C. The results of these tests are presented in Figure 42 for freezing fog, Figure 43 for snow, and Figure 44 for light freezing rain. Figure 42 shows that the fluid F4A holdover time varies from 78 to 105 minutes under the same freezing fog testing conditions, depending on whether the fluid had been shaken or not. The two other fluids did not demonstrate a significant variation of the holdover time with the shaking of the fluid. When a fluid was applied warm, it was shaken before application on only one of the two test panels. However, both panels presented the same endurance time for the three fluids tested.

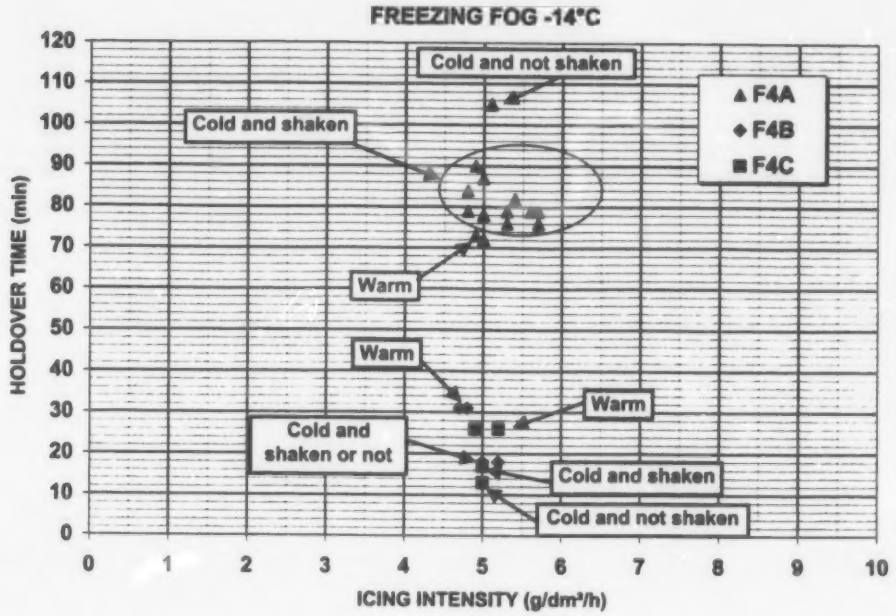


Figure 42: Fluid Application: Freezing Fog Tests

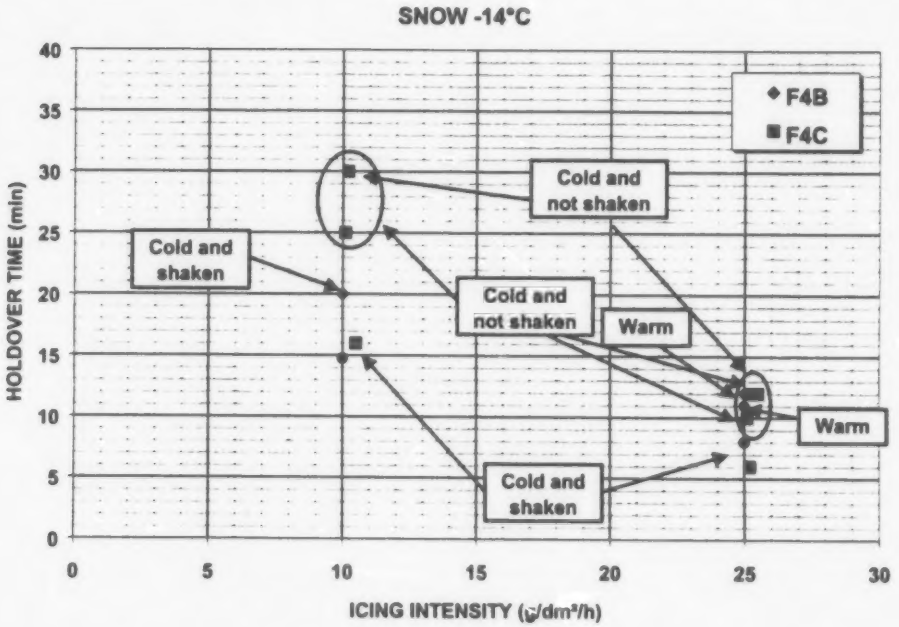


Figure 43: Fluid Application: Snow Tests

Figure 43 shows that the holdover time for F4B can vary from 15 to 25 minutes and for F4C from 20 to 30 minutes under the same environmental conditions of snow tests, depending on whether the fluid has been shaken or not. When the fluids were tested warm, their endurance time was not influenced by the shaking. It is interesting to note that the anti-icing endurance time of these fluids was not influenced by the shaking when they were tested under freezing fog conditions.

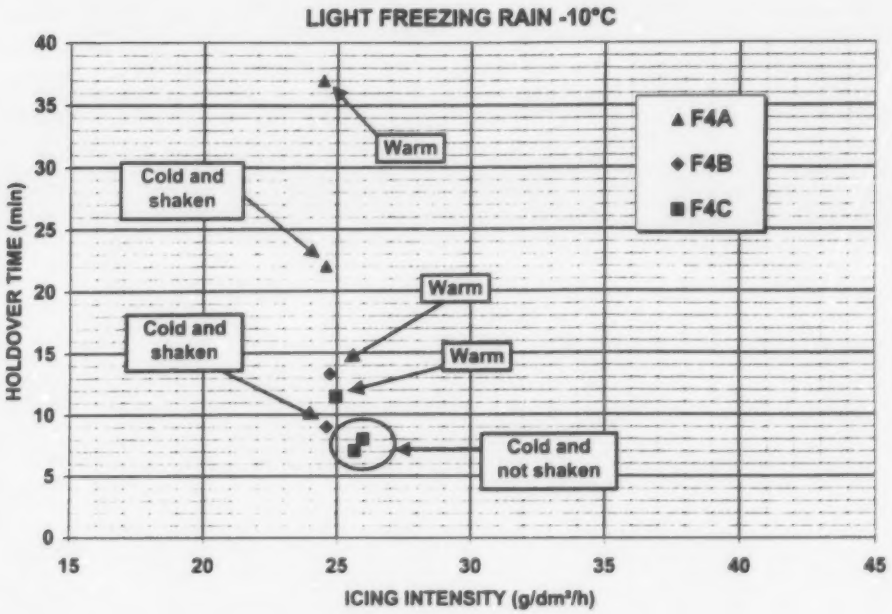


Figure 44: Fluid Application: Light Freezing Rain Tests

Figure 44 shows how the anti-icing endurance times for the three fluids are affected by the temperature of application under the light freezing rain condition. A comparison of fluid endurance times obtained under three different types of precipitation is presented in Table 7 and shows the influence of the fluid temperature at application time on the anti-icing endurance time. It seems difficult to identify a clear pattern of behaviour. While under freezing fog and light freezing rain conditions, the short cold endurance times (under

seems difficult to identify a clear pattern of behaviour. While under freezing fog and light freezing rain conditions, the short cold endurance times (under 20 minutes) increased when the fluid was applied warm, under snow precipitation they remained the same, at around 10 minutes. Therefore, the results of the comparison are inconclusive.

Table 7: Test Summary – Cold vs. Warm Fluid

Fluid Code	Precipitation Type	Temperature (°C)	Icing Intensity ($g/dm^2/h$)	Cold Fluid Endurance Time (min)	Warm Fluid Endurance Time (min)
F4A	Freezing fog	-14	5	76 to 115	73
F4B	Freezing fog	-14	5	18	31
F4C	Freezing fog	-14	5	13	26
F4A	Light freezing rain	-10	25	10 to 22	37
F4B	Light freezing rain	-10	25	7.5	13.5
F4C	Light freezing rain	-10	25	7.5	11.75
F4B	Snow	-14	25	10	12
F4C	Snow	-14	25	12	11

However, the fluid application at a warm temperature, 20 ± 5°C, was adopted at the SAE Fluids Subcommittee meeting organized by Transport Canada in Montreal in March 1999, since all the tests performed showed consistent results whether the fluid was shaken or not when it was applied warm, and because the fluid application procedure described in AMS1428C calls for a fluid applied warm. Furthermore, it was decided at the same meeting to extend the conformance to AMS1428C procedure by including a five-minute settling time after fluid application on the test panel before submitting the fluid to precipitation.

7.9 Fluid X Snow Tests

To compare various snow generation systems, a fluid of known formula was provided by the Union Carbide Corporation to generate a reference anti-icing endurance time table. This standard fluid was named Fluid X.

The formulation of Fluid X is presented in Table 8.

Table 8: Standard Fluid X Formula

Quantity	Component
650 g	Ethylene Glycol
350 g	Deionized Water
6.0 g	Food Grade Xanthan Gum*

* viscosity: 1200-1600 mPa.s
transmittance: not more than 85 percent
particle size: 100 percent through 60 mesh (250 μm) and at least 95 percent through 80 mesh (180 μm)

The procedure to make fluid X is as follows:

1) Pour ethylene glycol into a container;
2) Slowly add the Food Grade Xanthan Gum while mixing;
3) Slowly add water while mixing;
4) Agitate for at least 20 minutes, until a homogeneous mixture is obtained.

The Fluid X characteristics are presented in Table 9.

Table 9: Standard Fluid X Characteristics

Characteristics	T (°C)	Value	Tolerance
Refractive Index	20	1.3969	± 0.0006
pH	20	6.56	± 0.04
Viscosity* (mPa.s)	20	40 200	± 21.6%

* Brookfield, 0.3 rpm, cylindrical spindle #2, or equivalent

Snow tests were performed with Fluid X under two fluid application procedures. The results of the tests are presented in Table 10. The first procedure resulting in the cold fluid endurance times was used for all previous tests, in which the fluid was applied cold, except Fluid X was not sheared. For the second procedure resulting in warm fluid endurance times, Fluid X was sheared less than two hours before the test and applied warm more than twenty minutes after shearing. The fluid was left to settle on the test panel for five minutes before being submitted to precipitation. The latter procedure was adopted at the March 1999 meeting.

Table 10: Standard Fluid X Anti-icing Endurance Times in Snow Simulation Tests

Temperature (°C)	Icing Intensity ($g/dm^2/h$)	Cold Fluid Endurance Time (min)	Warm Fluid Endurance Time (min)
-3	10	45	59
-3	25	22	30
-14	10	35	35
-14	25	18	16
-25	5	65	50
-25	10	35	25

As can be seen in Table 10, the cold fluid endurance times are shorter at -3°C, similar at -14°C, and longer at -25°C than the warm fluid endurance times. A fluid fails when its temperature and dilution reach its freezing point. The initial temperature of the fluid influences its temperature during the test. Therefore, if a fluid is warmer at the beginning, it requires a higher level of dilution to reach a frozen state. The initial temperature also affects the fluid viscosity, and therefore the fluid thickness on the test panel. Consequently, if a fluid is warmer, hence less viscous, it will be thinner on the panel and will reach the freezing dilution sooner. Therefore, a warm fluid will induce two opposite effects regarding the failure time, and it is difficult to predict which effect will be the most influential.

8. CONCLUSION

Following an SAE G-12 committee recommendation, a series of procedures for the determination of anti-icing fluid holdover time guideline tables was developed. These procedures were mostly based on AMS1428C Annex A, which is the SAE document for anti-icing fluid certification, and on the resolutions adopted at the meeting of the Workgroup on Laboratory Methods to Derive Holdover Time Guidelines [4].

The set of tests required to build a holdover time table must include each type of precipitation and cover the overall range of temperatures. The types of precipitation are: frost, freezing fog, snow, freezing drizzle, light freezing rain, and rain on a cold-soaked wing. The air temperature values for the tests are: 0, -3, -14, -25°C, and lowest operational use temperature (LOUT) if it is below -25°C for frost; -3, -14, -25°C and LOUT for freezing fog and snow; -3 and -10°C for freezing drizzle and light freezing rain; and 1°C for rain on a cold-soaked wing. For frost and for all tests at LOUT, fluids are tested at only one icing intensity at each temperature; but for all other types of precipitation and for temperatures other than LOUT, fluids are tested at two icing intensities: a moderate intensity and a high intensity. The former yields the high number of the corresponding cell in the holdover time table and the latter yields the low number. The icing intensities vary for each type of precipitation. Table 11 shows the icing intensities selected for frost. For tests at LOUT the icing intensity was not determined and will be reported as measured. Table 12 shows the moderate and high intensities selected for all types of precipitation but frost.

Table 11: Frost Icing Intensities

Air Temperature (°C)	0	-3	-14	-25	LOUT
Icing Intensity (g/dm²/h)	0.20	0.20	0.13	0.05	0.05

Table 12: Icing Intensities for All Types of Precipitation Except Frost

Icing Intensity Level	Freezing Fog	Snow	Freezing Drizzle	Light Freezing Rain	Rain on a Cold-Soaked Wing
Moderate	2	10*	5	13	5
High	5	25*	13	25	75
LOUT	5	5	-	-	-

* Exception: at -25°C for snow, moderate is 5, high is 10.

All tests were performed in climatic chambers with specialized equipment. Different types of liquid precipitation require different sizes of nozzle. Freezing fog is simulated using a humidification system and a frosticator able to maintain the test panels at a temperature 3°C lower than the ambient air temperature during the test. For the rain on a cold-soaked wing simulation, a cold soak box is used. A snow generation system was specially conceived and built for the snow tests. It consists of an automated distribution mechanism capable of applying snow evenly over a test panel. The artificial snow is constituted of agglomerates of tiny (about 100 μm) frozen droplets.

For all tests but frost tests, the fluid test panels are 500 mm long, 300 mm wide, and 3.2 mm thick, and their temperature is that of the ambient air at the beginning of the test, but is free to vary during the test. For frost, the panels are 300 mm long, 100 mm wide, and 1.6 mm thick, and their temperature is maintained at a prescribed temperature by a special cooling system.

Calibration tests are performed to measure the average and distribution of the icing intensity over each test panel. For each 500 mm x 300 mm test panel, at least twelve 100 x 100 mm pans or plates are used to collect the ice, and for each 300 mm x 100 mm test panel, three plates are used. The average weight and standard deviation of the ice collected by the calibration pans or plates allow calculation of the average and standard deviation of the icing intensity. Generally, the average icing intensity and the standard deviation must remain

respectively within 4 and 6 percent of the target intensity. The tolerance is increased when the target intensity is small.

The tolerance on the average air temperature is usually ± 0.5°C, except at -25°C and at LOUT, when it is increased to 2.0°C. The tolerance on the air temperature standard deviation is usually 0.2 or 0.3°C, except at -25°C and at LOUT, when it is increased to 0.5°C.

Over four hundred tests, including calibration and fluid tests, were conducted under all types of precipitation conditions. The tests verified that the test parameter tolerances defined in the previous section could easily be met. The fluid tests also identified problems in the test procedures, such as the lack of reproducibility due to variations in fluid texture at the time of application. A March 1999 meeting led to a decision to modify the application procedure and to adopt that of the current AMS1428C anti-icing endurance tests. Snow tests were performed with a standard fluid, fluid X, formulated by the Union Carbide Corporation, to establish a reference to snow generation system calibration.

As a follow-up to the work presented in this report, a procedure for each precipitation type was written and proposed for an SAE ballot. At the SAE March 1999 meeting, the results of the ballot were discussed and the procedures amended. The modified procedures, which were again submitted for ballot in May 1999, are reproduced as appendices to this report.

REFERENCES

[1] **SAE Standard** AMS 1428C: *Fluid, Aircraft Deicing/Anti-icing, Non-Newtonian (pseudoplastic), SAE Types II, III and IV*, October 1997.

[2] **Bernardin, S., Dubuison, C., and Laforte J.L.** (1997) *Development of Laboratory Test Procedures to Replace Field Anti-icing Fluid Tests (Snow Equivalence Tests).* Report prepared for the Transportation Development Centre, TP3141E, November 1997, 110 pages.

[3] **Dawson, P., D'Avirro, J., and Potter, R.V.** (1996) *Validation of Methodology for Simulating a Cold-Soaked Wing.* Report prepared for the Transportation Development Centre, TP12899E, October 1996, 92 pages.

[4] **SAE Minutes of Meeting:** *Workgroup on Laboratory Methods to Derive Holdover Time Guidelines.* 20-21 November 1997.

[5] **Rasmussen, R., Knight, C., and Hills, A.** (1999) *Development of a Method to Test Holdover Times of Deicing and Anti-icing Fluids in a Cold Room Using Artificially Generated Snow.* Report prepared for the FAA, FAA/AR – 98/74, January 1999, 21 pages.

APPENDICES

APPENDIX FOG

PROCEDURES FOR ANTI-ICING ENDURANCE TIME TESTING OF AIRCRAFT DEICING/ANTI-ICING FLUID UNDER FREEZING FOG CONDITIONS

1. SCOPE

1.1 This document establishes the minimum requirements for an environmental test chamber and test procedure to carry out freezing fog anti-icing endurance time tests according to the current materials specification for aircraft deicing/anti-icing fluids. The primary purpose for such a test method is to determine freezing fog anti-icing endurance times under controlled laboratory conditions for SAE Type II, Type III, and Type IV fluids.

1.2 While the materials, methods, applications, and processes described or referenced in this appendix may involve the use of hazardous materials, this appendix does not address the hazards that may be involved in such use. It is the sole responsibility of the user to ensure familiarity with the safe and proper use of any hazardous materials and processes, and to take necessary precautionary measures to ensure the health and safety of all personnel involved.

1.3 The values stated in SI units are to be regarded as the standard.

2. REFERENCED DOCUMENTS

2.1 SAE Publications:

Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001.

AMS 4037 Aluminum Alloy Sheet and Plate, 4.4 Cu - 1.5 Mg - 0.6 Mn (2024-T3 Flat Sheet, T351 Plate) Solution Heat Treated

2.2 ASTM Publications:

Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

ASTM D 1193 Reagent Water

2.3 ISO Publication:

Available from International Organization for Standardization, Case Postal #56, rue Varembe, CH-1211, Switzerland.

ISO 10012: Quality assurance requirements for measuring equipment

3. SUMMARY OF TEST

3.1 This test permits the determination of the laboratory freezing fog anti-icing endurance times of SAE Type II, Type III, and Type IV fluids. The fluids to be evaluated are applied to a test plate exposed to freezing fog conditions, and their anti-icing endurance time is evaluated by measuring the minimum exposure time before a specified degree of freezing occurs. A general description of the anti-icing endurance time tests referred to in this appendix is as follows.

3.2 Freezing Fog Simulation Test:

This test involves pouring the fluid onto a test plate inclined at a 10° angle from the horizontal. The anti-icing endurance time is recorded as the time for ice formation to cover 30% of the test plate. Seven tests corresponding to different sets of temperature and icing intensity are produced:

Table 1: Freezing Fog Test Conditions

Test Condition	Air Temperature (°C)	Icing Intensity ($g/dm^2/h$)
FOG-A	-3	2
FOG-B	-3	5
FOG-C	-14	2
FOG-D	-14	5
FOG-E	-25	2
FOG-F	-25	5
FOG-G	<-25*	5

** This test will be performed at the lowest usable temperature of the fluid if it is below -25°C.*

It is a fundamental requirement of this test that the spray impinges onto the surface of the test plate as supercooled water droplets that freeze on impact. This is verified by observation of an untreated (no fluid) ice catch plate.

4. EQUIPMENT AND TEST PARAMETERS

4.1 General:

A description of the minimum requirements for the environmental test chamber and associated test equipment, including test plate, spray equipment, and data acquisition, is given below. A summary of the performance requirements for the test equipment is given in Table 2. Other spray equipment that meets the requirements of this appendix is acceptable.

Table 2: Summary of Performance Requirements for Test Equipment

Test Parameters	Requirements
Test Chamber	
• Minimum volume	7 m^3 for each 300 mm x 500 mm test plate
• Air temperature range	0 to at least -25°C
• Average set point temperature	± 0.5°C
• Standard deviation	0.3°C at -3°C and -14°C 0.5°C at -25°C and below -25°C
• Minimum temperature sampling rate	1 datum per minute
• Horizontal air velocity	≤ 0.4 m/s
• Relative humidity	> 40% Rh
Test Plate	
• Material	Aluminum alloy AMS 4037
• Surface finish	Average surface roughness Ra = 0.1 to 0.2 µm
• Test panel dimensions	500 mm long x 300 mm wide x 3.2 mm thick
• Slope	10° ± 0.2
• Temperature at start of test	Within 0.5°C of air temperature
Ice Catch Plates	
• Reference ice catch plate dimensions	100 mm x 100 mm x 1.6 mm thick
• Minimum number of reference ice catch plates per test plate	8
Spray Equipment	
• Water supply to nozzle	conforming to ASTM D1193 Type IV
• Water supply temperature	< 2°C measured just before the nozzle
• Water droplet size	22 ± 5 µm MVD water droplets
• Water spray intensity	2.0 g/dm²/h ± 0.2 *and* 5.0 g/dm²/h ± 0.2

4.2 Test Chamber (see Table 2):

The plate and the air temperature shall be within 0.5°C prior to starting the test. The temperature sensing device shall be in proximity to the test plate, typically within 1.5 m of the side of the test plate, but outside the spray area. The distance and position of the sensing device shall be reported. The air exchange rate in the chamber shall correspond to an air velocity no greater than 0.4 m/s when measured 50 mm above the surface of the test plate. The test chamber shall be equipped with artificial lighting such that it does not interfere with the precipitation nor with the air, fluid, and plate temperatures.

The air temperature, plate temperature, and humidity sensing devices shall be linked to an electronic data acquisition system as a means of checking the environmental control characteristics of the test chamber throughout the course of a test run.

4.3 Test Plate (see Table 2):

Each test plate is a removable panel placed on a support housed within the test chamber. Each panel shall be marked with a horizontal line 150 mm from the upper shorter edge of the test plate or with any other permanent pen marking used to estimate the degree of ice formation on fluid-treated panels during the course of a test run.

Each test plate shall be equipped with a temperature sensor located on the underside of the plate. This sensor shall be capable of measuring to an accuracy of ± 0.5°C and shall be linked to an electronic data acquisition system.

The test plate support shall be set up in such a way that it can accommodate a minimum of eight 100 mm x 100 mm ice catch plates surrounding, but not in contact with, each test plate (see Figure 1). The test plate support face shall be inclined at a 10° angle ± 0.2 from the horizontal. The test plates shall be placed on the support such that the fluid can freely flow off all edges of the plate. The test stand shall be designed to minimize the contact between the test surface and the support.

4.3.1 Precipitation Catch Plates:

The ice catch plates described in Table 2 are used to evaluate the ice catch, as described in section 5.4.

4.4 Spray Equipment:

4.4.1 General Requirements:

The equipment used to provide the water spray includes a low flow nozzle supplied with water conforming to ASTM D1193 Type IV. This equipment is housed in the upper region of the test chamber above the test plate.

The exact type and geometry of the spray system used to generate the water spray for the test is left to the discretion of the user, provided the following parameters are met.

(a) Median volume diameter of the water spray droplets shall be 22 ± 5 μm.

(b) The average intensity of the water spray produced during a test shall correspond for conditions FOG-A, FOG-C, and FOG-E: 2 g/dm^2 ± 0.2 per hour, and for conditions FOG-B, FOG-D, FOG-F, and FOG-G: 5 g/dm^2 ± 0.2 per hour.

(c) The water spray shall be evenly distributed over the entire area of the test plate. Even distribution is verified by the standard deviation obtained from a calibration test described in section 5.4.

(d) The water spray shall impinge on the surface of the test plate in the form of supercooled water droplets at a temperature close to that of the ambient air temperature.

To provide some background information, an example of a suitable spray system is outlined below.

4.4.2 Example of Spray Equipment:

The nozzle comprises two sections: outer and inner units for the respective passage of water and compressed air. The critical dimensions are given in Figure 2. The nozzle reciprocates and can provide even and reproducible coverage of the test plate at the specified rate.

4.5 Temperature Control Equipment:

The air temperature shall be maintained at the required level using heat exchangers connected to temperature control equipment featuring a solid state temperature sensor such as a platinum resistance probe (100 ohms at 0°C) coupled to a proportional temperature controller having a minimum resolution of 0.5°C.

4.6 Air Distribution System:

This system shall consist of a fan or fans to provide air recirculation through the main body of the test chamber and to the heat exchanger. Ducting for the passage of air at both the inlet and the outlet of the heat exchanger shall have entry and exit ports positioned to provide good air recirculation throughout the test chamber. The heat exchanger shall be capable of cooling the air and maintaining it at the specified temperature level. Air flow shall be measured using a suitable anemometer or velometer.

5. CALIBRATION OF TEST EQUIPMENT

5.1 Standard Measuring Devices:

All temperature sensors, humidity sensors, electronic balances, anemometers, and timing devices shall be maintained in a known state of calibration in accordance with recognized international standards such as ISO 10012, by calibrating each instrument every six months or whenever a piece of equipment is repaired, replaced, or moved, and a written record of each calibration shall be kept available.

5.2 Average Air Velocity:

The average horizontal component of the air velocity in the test chamber, when measured 50 mm above the centerline at the upper edge of the test plate, must not exceed 0.4 m/s.

5.3 Water Droplet Size Determination:

Several methods are available to determine the water droplet sizes referred to in Table 2. The following are acceptable examples:

(a) Slide Impact Method with Oil: A sample of the water droplets from the precipitation is collected on an oil-coated microscope slide. An oil having a viscosity of 5000 mPas at 20°C, spread to a thickness of about 500 µm, will be suitable. The oil can be either a mineral oil or a silicone oil. The droplet size is determined by direct observation under a microscope using an eyepiece with the appropriate graticle, or from enlarged photographs of the slide.

(b) Slide Impact Method with Colloidal Silver: A sample of the water droplets from the precipitation is collected on a microscope slide coated with a colloidal silver solution. A thin film of 95% water and 5% colloidal silver is brushed over a microscope slide. Once the film is dry (about 30 seconds), it is exposed to the water spray, where it becomes permanently marked with the droplet imprints. The droplet diameter is one third of the print diameter measured under a microscope using an eyepiece with the appropriate graticle, or from enlarged photographs of the slide.

(c) Laser Diffraction Method: Using a laser diffraction particle analyzer incorporating a low power laser transmitter and photo detector, the size of the droplets can be measured as they fall toward the surface of the test plate. This is done by analyzing the diffraction patterns, which will give the size and the distribution of the droplets. Some equipment is capable of achieving this in real time.

5.4 Ice Catch Calibration:

For each anti-icing endurance time test, it is important to establish that even and reproducible ice formation occurs over the surface of the test plates. To carry out this evaluation, ice catch measurements shall be performed under each test condition. A summary of the test conditions follows.

Table 3: Ice Catch Calibration for the Different Test Conditions

Condition	FOG-A	FOG-B	FOG-C	FOG-D	FOG-E	FOG-F	FOG-G
Air Temperature (°C)	-3 ± 0.5	-3 ± 0.5	-14 ± 0.5	-14 ± 0.5	-25 ± 1	-25 ± 1	<-25 ± 1**
Water Spray Intensity ($g/dm^2/h$)	2.0 ± 0.2	5.0 ± 0.2	2.0 ± 0.2	5.0 ± 0.2	2.0 ± 0.2	5.0 ± 0.2	5.0 ± 0.2
Standard Deviation ($g/dm^2/h$)	0.2	0.3	0.2	0.3	0.2	0.3	0.3
Calibration Test Duration (min)	30 and 120	30 and 120	30 and 120	30 and 120	30 and 120	30 and 120	30 and 120

** *This test will be performed at the lowest usable temperature of the fluid if it is below -25°C.*

To assess the ice catch for each condition, a total of at least 20 100 mm x 100 mm ice catch plates are used. Each test panel is replaced with 12 ice catch plates, which in turn are surrounded by at least 8 additional reference ice catch plates (see Figure 3). These preweighed plates are weighed upon completion of each test and the difference in the recorded weights is the ice catch for that plate. The average ice catch on the plates replacing the test panel is calculated as well as the average ice catch of the reference plates. It is the ratio between these two values that is used to estimate the icing intensity during a fluid test run when only the reference plates are available, i.e.:

from calibration test: $$Ratio = \frac{I_{plate}}{I_{ref}}$$

where:

$Ratio$ = ratio of the ice catch over the plates replacing the test panel with respect to the ice catch on the reference plates

I_{plate} = the average ice catch on the plates replacing the test panel

I_{ref} = the average ice catch on the reference plates

During an anti-icing endurance time test run, the ice catch is measured on the reference plates, averaged, and this value is then multiplied by the ratio calculated above based on a calibration test performed under the same conditions. The resulting value is the estimated icing intensity over the test panel:

for fluid test run: Estimated $I_{plate} = Ratio \times I_{ref}$

where I_{ref} is measured during a fluid test and *Ratio* has been determined in a previous calibration test.

For a calibration test to be valid, the ice catch on the plates replacing the test panel shall fall within the variation specified in Table 3.

This calibration test shall be run at least every six months or whenever a piece of equipment is repaired, replaced, or moved.

6. TEST PROCEDURE

6.1 Test Plate Cleanliness:

The test plates shall be free of all visible contamination, smears, or stains, except for the horizontal line marked at the 150 mm point from the upper edge of the test plate or any other marking used to estimate ice coverage. Between test runs, any contamination shall be removed by washing with hot water immediately followed by ethanol. If the same fluid is tested on the same plate for two consecutive tests, it is not necessary to clean the plates with ethanol before the second test, hot water is sufficient.

6.2 Prepare 500 mL of the candidate test fluid for each test panel to be coated. If more fluid is required, the quantity of fluid actually used shall be mentioned in the report qualification statement. The fluid shall be sheared in accordance with the specification for the respective SAE Type fluid. The test shall start within two hours after the product has been sheared, but not within the first 20 minutes after shearing.

6.3 Ensure the test chamber and the fluid are at the required temperature. Verify that the test plate temperature is within 0.5°C of the air temperature. Fluids shall be applied at 20°C ± 5.

6.4 Pour the fluid onto each test plate, start the timing device, cover each test plate with a removable cover, and turn on the water spray. After 5 minutes, remove the cover from each test plate. Observe the panels and, when failure occurs as defined in section 6.5, record the time as the anti-icing endurance time. When the water spray is turned off, weigh the ice catch on each 100 mm x 100 mm reference ice catch plate and, using the method described in section 5.4, estimate the icing intensity for each test panel. If the icing intensity is within the specified limits for the test being conducted, the time recorded is valid for the test.

Delayed crystallization may occur during the course of a test run, and can be easily recognized by a sudden (within 30 seconds) frozen contamination coverage of a large surface of the plate. If this coverage exceeds 30% of the plate's total surface, the test is invalid and must be repeated. In the case of suspected delayed crystallization, if the test has been invalidated 3 times, the plate may be seeded at its center top edge with an ice crystal to initiate crystallization. Seeding consists of putting an ice crystal in contact with the fluid by means of a chilled metal rod (below 0°C). If the ice suddenly covers more than 30% of the seeded plate's surface, the test is invalid.

6.5 Failure Criterion:

Failure is called when 30% of the plate is covered with frozen contamination. Examples of the appearance of this frozen contamination include, but are not limited to:

- Ice front
- Ice sheet
- Slush, in clusters or as a front
- Disseminated fine ice crystals

- Frost on surface
- Clear ice pieces partially or totally imbedded in fluid

Pen marks on the plate can be used to estimate the area of failure. For instance, a line drawn across the plate at 150 mm from the top edge will delineate an area corresponding to 30% of the plate.

6.6 Reproducibility/Precision:

The freezing fog test is dynamic by nature, and small variations can be expected. Each test condition shall be tested on at least two panels. If the maximum variation of the anti-icing endurance time is less than 10% of the average anti-icing endurance time, report this time. If not, repeat testing on two additional panels, for a total of four data points. The highest and lowest points shall be discarded, and the average of the two remaining points shall be the anti-icing endurance time for that condition.

6.7 Report:

The report shall state the name and address of the facility conducting the tests, together with a statement confirming the test facility is autonomous of the manufacturer or vendor of the fluid. The following information shall also appear on the test document:

Date tests conducted
Manufacturer or vendor's name and address
Name or reference number and lot number of the fluid tested
Type of fluid (SAE Type II, SAE Type III, or SAE Type IV)
Summary of test results, and the ice catch results for each test
Method of estimation of failure area
Description of appearance of frozen contamination (section 6.5)

Condition of fluid: Concentrate as supplied to test facility, or diluted with hard water as defined in the specification and subsequently sheared in accordance with specification giving the dilution as a ratio (example: SAE Type II diluted 75/25 with water).

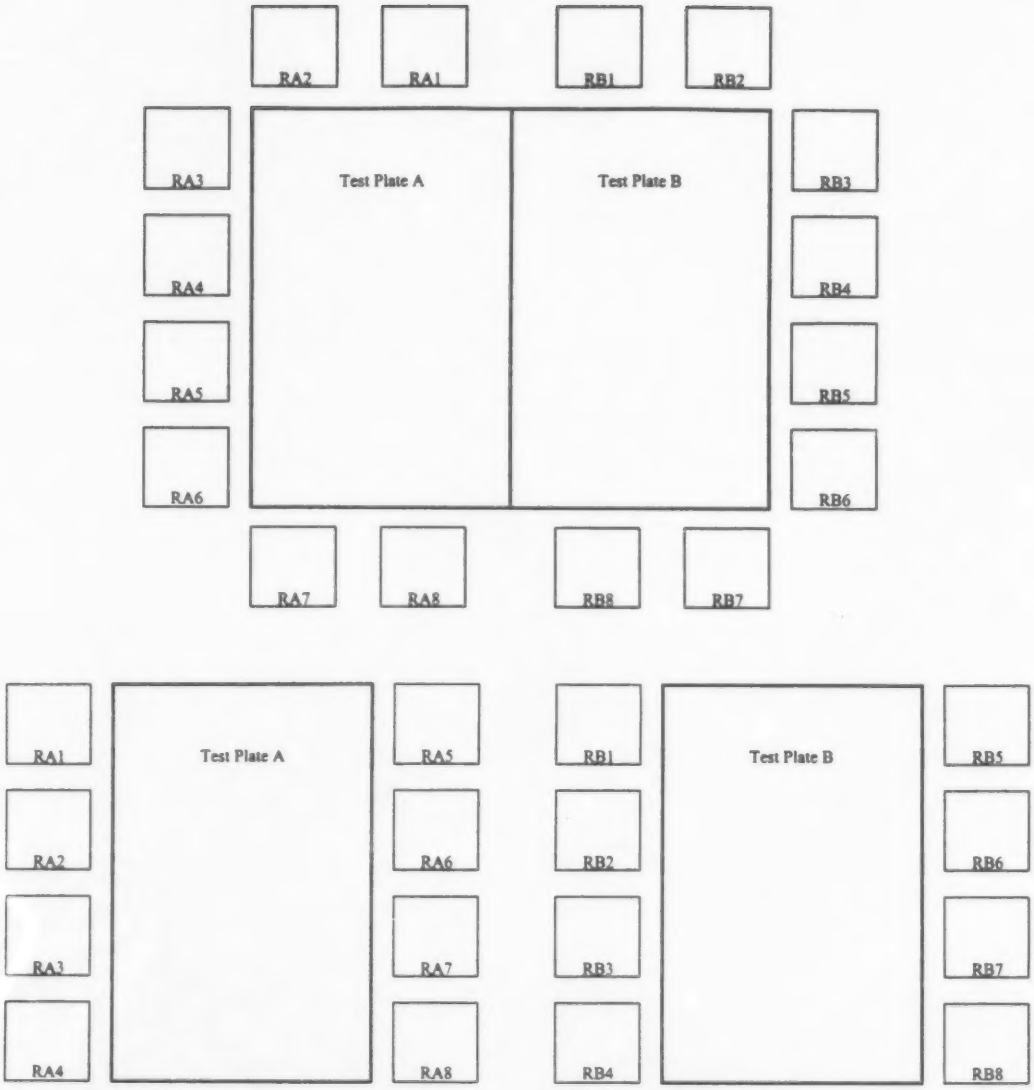


FIGURE 1 – Two examples where two test plates are each surrounded by 8 reference ice catch plates, used for the ice catch estimation

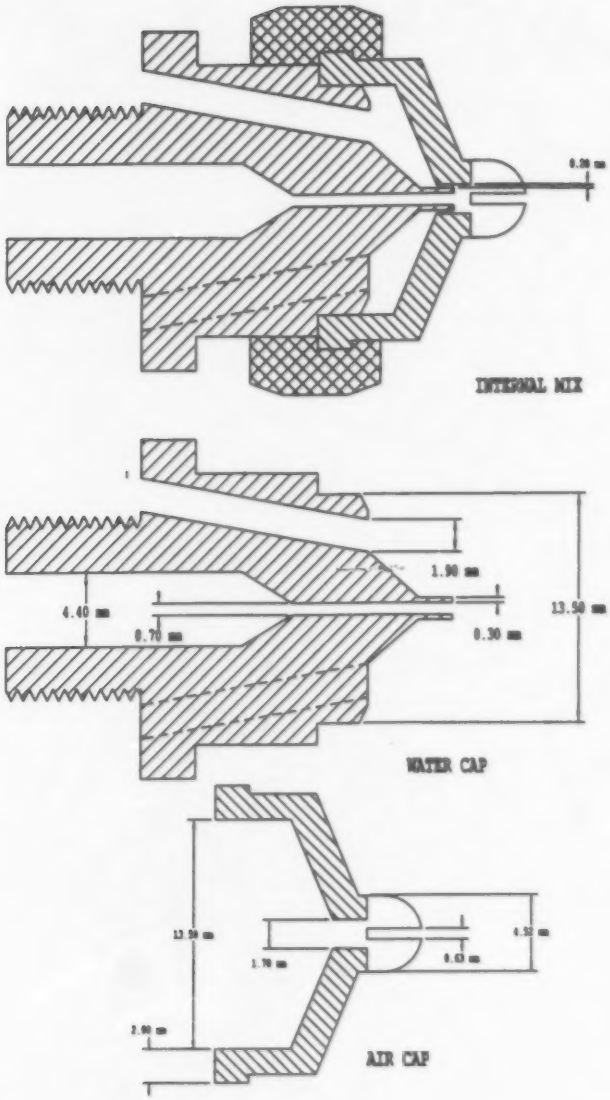


FIGURE 2 – Spray nozzle

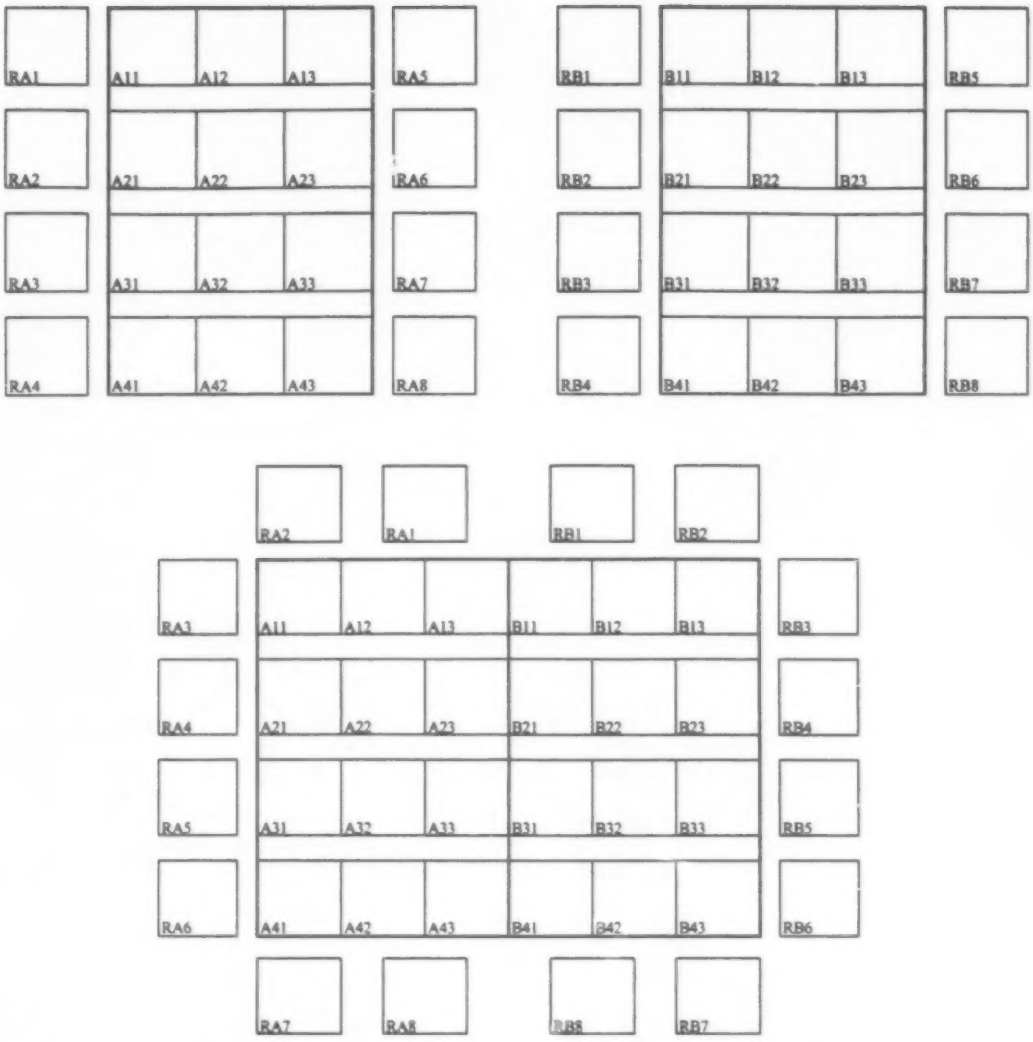


FIGURE 3 – Two examples of dispositions of reference ice catch plates (denoted RA1 to RA8 for plate A and RB1 to RB8 for plate B), each surrounding the 12 ice catch plates replacing the test plates (A11 to A43 for test plate A and B11 to B43 for test plate B)

APPENDIX ZL

PROCEDURES FOR ANTI-ICING ENDURANCE TIME TESTING OF AIRCRAFT DEICING/ANTI-ICING FLUID UNDER FREEZING DRIZZLE CONDITIONS

1. SCOPE

1.1 This document establishes the minimum requirements for an environmental test chamber and the test procedures used to carry out anti-icing endurance time tests of aircraft deicing/anti-icing fluids under freezing drizzle conditions. The primary purpose for such a test method is to determine the freezing drizzle anti-icing endurance time under controlled laboratory conditions for SAE Type II, Type III, and Type IV fluids.

1.2 While the materials, methods, applications, and processes described or referenced in this appendix may involve the use of hazardous materials, this appendix does not address the hazards which may be involved in such use. It is the sole responsibility of the user to ensure familiarity with the safe and proper use of any hazardous materials and processes, and to take necessary precautionary measures to ensure the health and safety of all personnel involved.

1.3 The values stated in SI units are to be regarded as the standard.

2. REFERENCED DOCUMENTS

2.1 SAE Publications:

Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001.

AMS 4037 Aluminum Alloy Sheet and Plate, 4.4 Cu - 1.5 Mg - 0.6 Mn (2024-T3 Flat Sheet, T351 Plate) Solution Heat Treated

2.2 ASTM Publications:

Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

ASTM D 1193 Reagent Water

2.3 ISO Publication:

Available from International Organization for Standardization, Case Postal #56, rue Varembe, CH-1211, Switzerland.

ISO 10012: Quality assurance requirements for measuring equipment

3. SUMMARY OF TEST

3.1 This test permits the determination of laboratory anti-icing endurance times of SAE Type II, Type III, and Type IV fluids. The test fluids to be evaluated are applied to a test plate exposed to freezing drizzle. Their anti-icing endurance times are evaluated by measuring

the minimum exposure time before a specified degree of freezing occurs. A general description of the anti-icing endurance time test referred to in this appendix is as follows.

3.2 Freezing Drizzle Simulation Test:

This test involves pouring the fluid onto a test plate inclined at a 10° angle from the horizontal. The anti-icing endurance time is recorded as the time for frozen contamination to cover 30% of the test plate. Four tests corresponding to different sets of temperature and icing intensities are produced:

Table 1: Freezing Drizzle Test Conditions

Test Condition	Air Temperature (°C)	Icing Intensity ($g/dm^2/h$)
ZL-A	-3	5
ZL-B	-3	13
ZL-C	-10	5
ZL-D	-10	13

It is a fundamental requirement of this test that the spray impinges onto the surface of the test plate as supercooled water droplets that freeze shortly after impact. This is verified by observation of an untreated (no fluid) ice catch aluminum pan.

4. EQUIPMENT AND TEST PARAMETERS

4.1 General:

A description of the minimum requirements for the environmental test chamber and associated test equipment, including test plate, spray equipment, and data acquisition, is given below. A summary of the performance requirements for the test equipment is given in Table 2. Other spray equipment that meets the requirements of this appendix is acceptable.

4.2 Test Chamber (see Table 2):

The plate and air temperature shall be within 0.5°C prior to starting the test. The temperature sensing device shall be in proximity to the test plate, typically within 1.5 m of the side of the test plate, but outside the spray area. The distance and position of the sensing device shall be reported. The air exchange rate in the chamber shall correspond to a horizontal air velocity of no greater than 1.0 m/s when measured 50 mm above the surface of the test plate. The test chamber shall be equipped with artificial lighting such that it does not interfere with the precipitation nor with the air, fluid, and plate temperatures.

The air temperature, plate temperature, and humidity sensing devices shall be linked to an electronic data acquisition system as a means of checking the environmental control characteristics of the test chamber throughout the course of a test run.

Table 2: Summary of Performance Requirements for Test Equipment

Test parameter	Requirements
Test Chamber	
• Distance between nozzle and plate	7 m ± 0.5
• Air temperature range	0 to -10°C
• Average set point temperature	± 0.5°C
• Standard deviation	0.3°C
• Minimum temperature sampling rate	1 datum per minute
• Horizontal air velocity	≤ 1.0 m/s
• Relative humidity	> 40%
Test Plate	
• Material	Aluminum alloy AMS 4037
• Surface finish	Average surface roughness: Ra = 0.1 µm to 0.2
• Test panel dimensions	500 mm long x 300 mm wide x 3.2 mm thick
• Slope	10° ± 0.2
• Temperature at start of test	Within ± 0.5°C of air temperature
Ice Catch Pans	
• Ice catch pan dimensions	100 mm x 100 mm x 0.8 mm thick with all-around rim 15 mm high
• Minimum number of reference ice catch pans per test plate	8
Spray Equipment	
• Water supply to nozzle	Conforming to ASTM D1193 Type IV
• Water supply temperature	< 2°C measured just before the nozzle
• Water droplet size and spray intensity	250 µm ± 50 median volume diameter for 5 g/dm²/h ± 0.2 and 350 µm ± 50 for 13 g/dm²/h ± 0.5

4.3 Test Plate (see Table 2):

Each test plate is a removable panel placed on a support housed within the test chamber. Each panel shall be marked with a horizontal line 150 mm from the upper shorter edge of the test plate or with any other permanent pen marking used to estimate the degree of ice formation on fluid-treated panels during the course of a test run.

Each test plate shall be equipped with a temperature sensor located on the underside of the plate. This sensor shall be capable of measuring to an accuracy of ± 0.5°C and shall be linked to an electronic data acquisition system.

The test plate support shall be set up in such a way that it can accommodate a minimum of eight 100 mm x 100 mm ice catch pans surrounding, but not in contact with, each test plate (see Figure 1). The test plate support face shall be inclined at a 10° angle ± 0.2 from horizontal. The test plates shall be placed on the support such that the fluid can freely flow off all edges of the plate. The test stand shall be designed to minimize the contact between the test surface and support.

4.3.1 Precipitation Catch Pans

The ice catch pans, described in Table 2 and Figure 2, are used to evaluate the ice catch, as described in section 5.4.

4.4 Spray Equipment:

4.4.1 General Requirements:

The equipment used to provide the water spray includes a low flow nozzle supplied with water conforming to ASTM D1193 Type IV. This equipment is housed in the upper region of the test chamber 7 m ± 0.5 above the test plate.

The exact type and geometry of the spray system used to generate the water spray for the test is left to the discretion of the user, provided the following parameters are met.

(a) Median volume droplet diameter of the water spray shall be:

ZL-A and ZL-C	250 ± 50 µm.
ZL-B and ZL-D	350 ± 50 µm

(b) The average intensity of the water spray produced during a test shall correspond to freezing drizzle conditions:

ZL-A and ZL-C	5 g/dm^2/h ± 0.2
ZL-B and ZL-D	13 g/dm^2/h ± 0.5

(c) The water spray shall be evenly distributed over the entire area of the test plate. Even distribution verified by the standard deviation obtained from a calibration test described in section 5.4.

(d) To allow the droplets to become supercooled at a temperature close to that of the ambient air temperature, the distance between the nozzle and the test plate shall be 7.0 m ± 0.5.

4.4.2 Example of Spray Equipment:

The hydraulic nozzle shown in Figure 3 comprises three sections: an outer unit holding two inner units. A prescribed volume of ASTM Grade IV water is stored in a pressurized tank and provides the flow to the nozzle. The water droplet size depends on the nozzle used, and a pulse system allows the flow of water through the nozzle to be controlled. The icing intensity is obtained by selecting the opening and closing times (on/off) of the pulse system. An even distribution over the test plate is achieved by a controlled oscillation of the nozzle.

4.5 Temperature Control Equipment:

The air temperature shall be maintained at the required level using heat exchangers connected to temperature control equipment featuring a solid state temperature sensor such as a platinum resistance probe (100 ohms at 0°C) coupled to a proportional temperature controller having a minimum resolution of 0.5°C.

4.6 Humidity Measuring Equipment:

This equipment consists of a humidity sensor of the capacitance, resistance, or conductivity type capable of covering a range of 40 to 100% RH from 0°C to -10°C.

4.7 Air Distribution System:

This system shall consist of a fan or fans to provide air recirculation through the main body of the test chamber and to the heat exchanger. Ducting for the passage of air at both

the inlet and the outlet of the heat exchanger shall have entry and exit ports positioned to provide good air recirculation throughout the test chamber. The heat exchanger shall be capable of cooling the air and maintaining it at the specified temperature level. Air flow shall be measured using a suitable anemometer or velometer.

5. CALIBRATION OF TEST EQUIPMENT

5.1 Standard Measuring Devices:

All temperature sensors, humidity sensors, electronic balances, anemometers, and timing devices shall be maintained in a known state of calibration in accordance with recognized international standards such as ISO 10012, by calibrating each measuring device at least once every six months or whenever a piece of equipment is repaired, replaced, or moved, and a written record of the calibrations shall be kept available.

5.2 Average Air Velocity:

The average horizontal component of the air velocity in the test chamber, when measured 50 mm above the centerline at the upper edge of the test plate, must not exceed 1.0 m/s.

5.3 Water Droplet Size Determination:

Several methods are available to determine the water droplet sizes referred to in Table 2. The following are acceptable examples:

(a) Slide Impact Method with Oil: A sample of the water droplets from the precipitation is collected on an oil-coated microscope slide. An oil having a viscosity of about 5000 mPas at 20°C, spread to a thickness of about 1000 µm, will be suitable. The oil can be either a mineral oil or a silicone oil. The droplet size is determined by direct observation under a microscope using an eyepiece with the appropriate graticle, or from enlarged photographs of the slide.

(b) Dye-Stain Method: The dye-stain technique consists of dusting filter paper discs with a water-activated, very finely divided powder form of methylene blue dye. The prepared discs are manually positioned under precipitation for a fixed time to acquire a droplet size pattern. A calibration curve is then used to convert from the measured diameter of the droplets on the pattern to the experimental median volume diameter.

5.4 Ice Catch Calibration:

For each anti-icing endurance time test, it is important to establish that even and reproducible ice formation occurs over the surface of the test plates. To carry out this evaluation, ice catch measurements shall be performed under each condition. A summary of the test conditions follows.

Table 3: Freezing Drizzle Simulation Test Ice Catch Calibration

Condition	ZL-A	ZL-B	ZL-C	ZL-D
Air Temperature (°C)	-3	-3	-10	-10
Mean Water Spray Intensity (g/dm²/h)	5 ± 0.2	13 ± 0.5	5 ± 0.2	13 ± 0.5
Standard Deviation (g/dm²/h)	0.3	0.7	0.3	0.7
Calibration Test Duration (min)	30 and 120	30 and 60	30 and 120	15 and 30

To assess the ice catch for each condition, a total of at least 20 100 x 100 mm ice catch pans are used. Each test plate is replaced with 12 ice catch pans, which in turn are surrounded by at least 8 additional reference ice catch pans (see Figure 4). These preweighed plates are weighed upon completion of each test and the difference in the recorded weights is the ice catch for that pan. The average ice catch on the pans replacing the test panel is calculated as well as the average ice catch on the reference pans. It is the ratio between these two values that is used to estimate the icing intensity during a fluid test run, when only the reference pans are available, i.e.:

from calibration test: $$Ratio = \frac{I_{plate}}{I_{ref}}$$

where:

$Ratio$ = ratio of the ice catch over the pans replacing the test plates with respect to the ice catch on the reference pans

I_{plate} = the average ice catch on the pans replacing the test plate

I_{ref} = the average ice catch on the reference pans

During an anti-icing endurance time test run, the ice catch is measured on the reference pans, averaged, and this value is then multiplied by the ratio calculated above based on a calibration test performed under the same conditions. The resulting value is the estimated icing intensity over the test panel:

for fluid test run: Estimated $I_{plate} = Ratio \times I_{ref}$

where I_{ref} is measured during a fluid test and $Ratio$ has been determined in a previous calibration test.

For a calibration test to be valid, the ice catch on the pans replacing the test plates shall fall within the variation specified in Table 3.

This calibration test shall be run at least once every six months or whenever a piece of equipment is repaired, replaced, or moved.

6. TEST PROCEDURE

6.1 Test Plate Cleanliness:

The test plates shall be free of all visible contamination, smears, or stains, except for the horizontal line marked at the 150 mm point from the upper edge of the test plate or any other marking used to estimate ice coverage. Between test runs, any contamination shall be removed by washing with hot water immediately followed by ethanol. If the same

fluid is tested on the same plate for two consecutive tests, it is not necessary to clean the plates with ethanol before the second test, hot water is sufficient.

6.2 Prepare 500 mL of the candidate test fluid for each panel to be coated. If more fluid is required, the quantity of fluid actually used shall be mentioned in the report qualification statement. The fluid shall be sheared in accordance with the specification for the respective SAE Type fluid. The test shall start within two hours after the product has been sheared, but not within the first 20 minutes after shearing.

6.3 Ensure the test chamber and fluid are at the required temperature. Verify that the test plate temperature is within 0.5°C of the air temperature. Fluids shall be applied at 20°C ± 5.

6.4 Pour the fluid onto each test plate, start the timing device, cover each test plate with a removable cover, and turn on the water spray. After 5 minutes, remove the cover from each test plate. Observe the panels and, when failure occurs as defined in section 6.5, record the time as the anti-icing endurance time. When the water spray is turned off, weigh the ice catch on each 100 mm x 100 mm reference ice catch pan and, using the method described in section 5.4, estimate the icing intensity for each test panel. If the icing intensity is within the specified limits for the test being conducted, the time recorded is valid for the test.

Delayed crystallization may occur during the course of a test run, and can be easily recognized by a sudden (within 30 seconds) frozen contamination coverage of a large surface of the plate. If this coverage exceeds 30% of the plate's total surface, the test is invalid and must be repeated. In the case of suspected delayed crystallization, if the test has been invalidated 3 times, the plate may be seeded at its center top edge with an ice crystal to initiate crystallization. Seeding consists of putting an ice crystal in contact with the fluid by means of a chilled metal rod (below 0°C). If the ice suddenly covers more than 30% of the seeded plate's surface, the test is invalid.

6.5 Failure Criterion:

Failure is called when 30% of the plate is covered with frozen contamination.
Examples of the appearance of this frozen contamination include, but are not limited to:

- Ice front
- Ice sheet
- Slush, in clusters or as a front
- Disseminated fine ice crystals
- Frost on surface
- Clear ice pieces partially or totally imbedded in fluid

Pen marks on the plate can be used to estimate the area of failure. For instance, a line drawn across the plate at 150 mm from the top edge will delineate an area corresponding to 30% of the plate.

6.6 Reproducibility/Precision:

The freezing drizzle simulation test is dynamic by nature, and small variations can be expected. Each test condition shall be tested on at least two panels. If the maximum variation of the anti-icing endurance time is less than 10% of the average anti-icing endurance time, report this time. If not, repeat testing on two additional panels, for a total of four data points. The highest and lowest points shall be discarded, and the average of the two remaining values shall be the anti-icing endurance time for that condition.

6.7 Report:

The report shall state the name and address of the facility conducting the tests, together with a statement confirming the test facility is autonomous of the manufacturer or vendor of the fluid. The following information shall also appear on the test document:

Date tests conducted
Manufacturer or vendor's name and address
Name or reference number and lot number of the fluid tested
Type of fluid (SAE Type II, SAE Type III or SAE Type IV)
Summary of the test results, and ice catch for each test
Method of estimation of failure area
Description of appearance of frozen contamination (section 6.5)

Condition of fluid: Concentrate as supplied to test facility, or diluted with hard water as defined in the specification and subsequently sheared in accordance with specification giving the dilution as a ratio (example: SAE Type II diluted 75/25 with hard water).

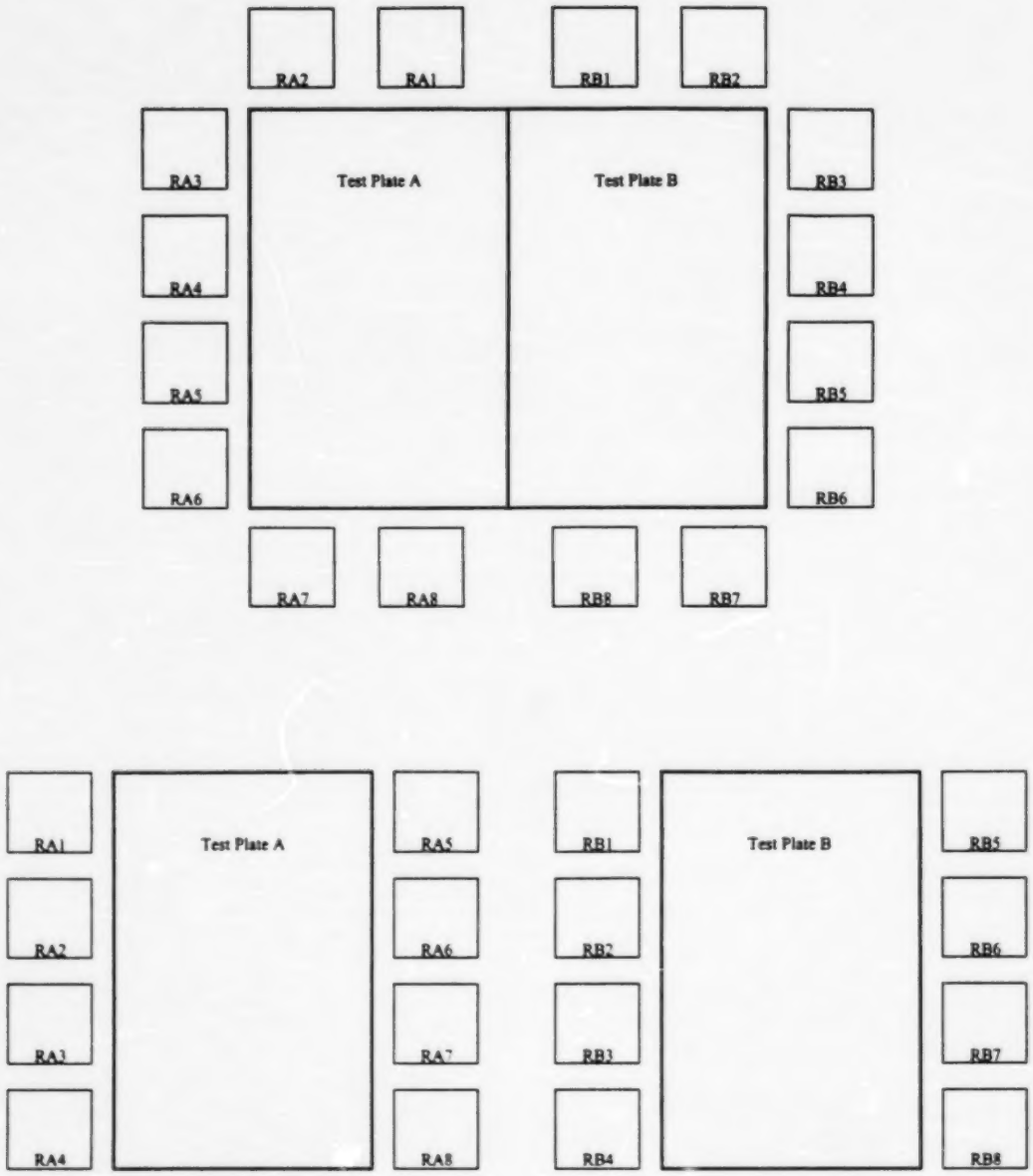


FIGURE 1 – Two examples where two test plates are each surrounded by 8 reference ice catch pans, used for the ice catch estimation

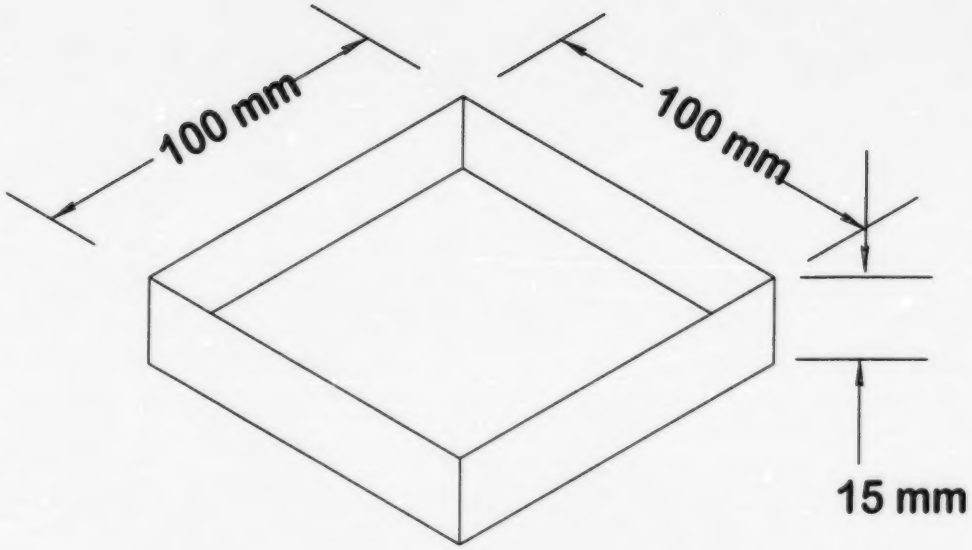


FIGURE 2 – Ice catch pan

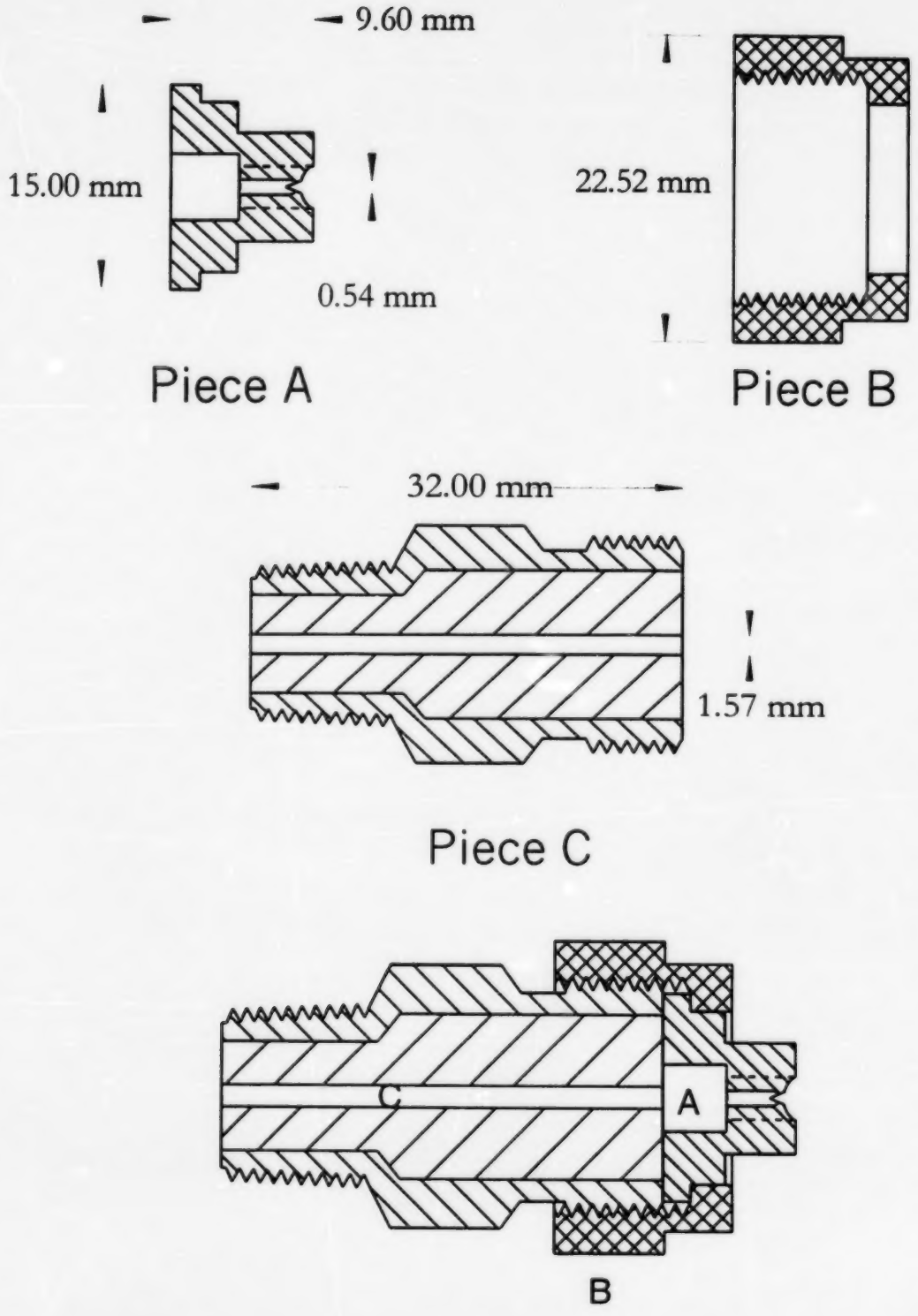


FIGURE 3 – Example of a hydraulic nozzle for freezing drizzle

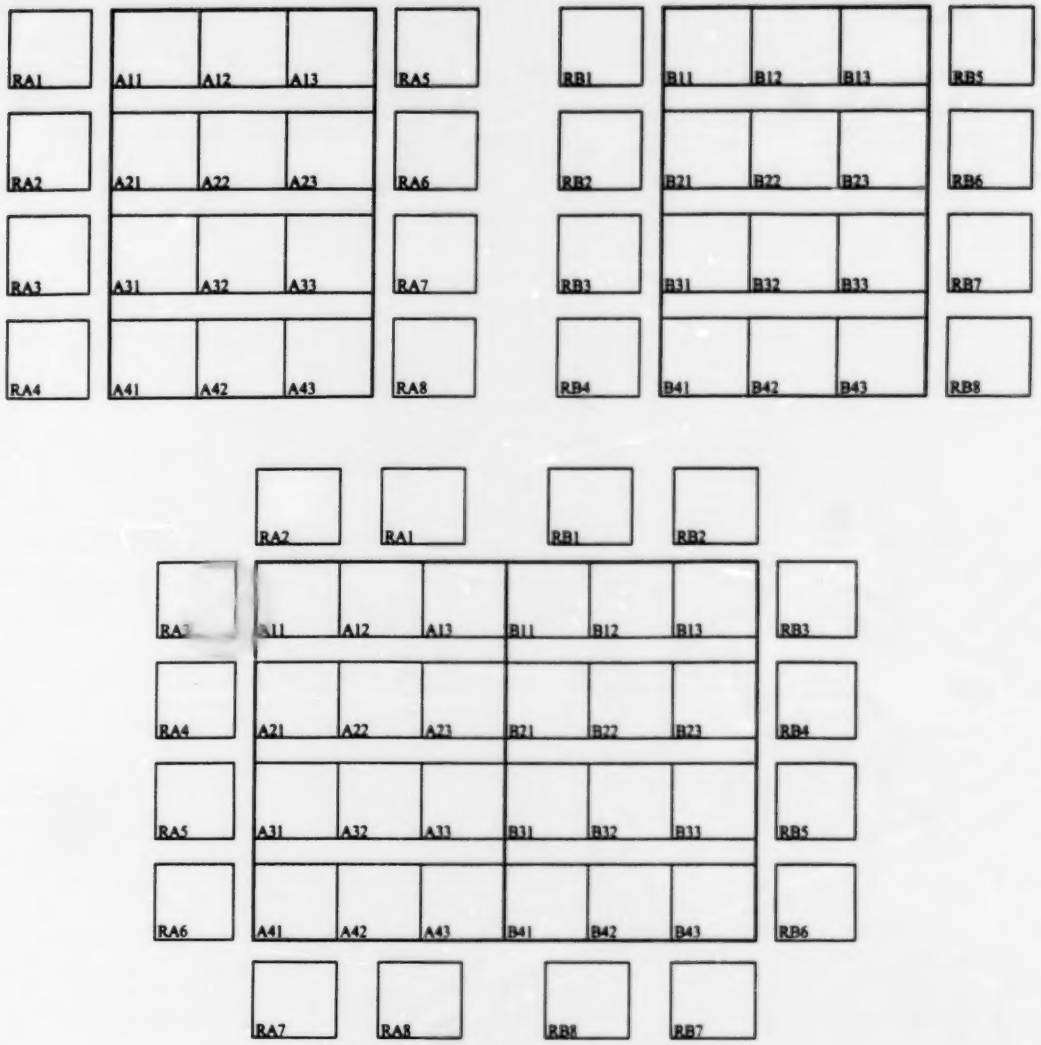


FIGURE 4 – Two examples of dispositions of reference ice catch pans (denoted RA1 to RA8 for plate A and RB1 to RB8 for plate B), each surrounding the 12 ice catch plates replacing the test pans (A11 to A43 for test plate A and B11 to B43 for test plate B)

APPENDIX LZR

PROCEDURES FOR ANTI-ICING ENDURANCE TIME TESTING OF AIRCRAFT DEICING/ANTI-ICING FLUID UNDER LIGHT FREEZING RAIN CONDITIONS

1. SCOPE

1.1 This document establishes the minimum requirements for an environmental test chamber and the test procedures used to carry out anti-icing endurance time tests of aircraft deicing/anti-icing fluids under light freezing rain conditions. The primary purpose for such a test method is to determine the light freezing rain anti-icing endurance time under controlled laboratory conditions for SAE Type II, Type III, and Type IV fluids.

1.2 While the materials, methods, applications, and processes described or referenced in this appendix may involve the use of hazardous materials, this appendix does not address the hazards that may be involved in such use. It is the sole responsibility of the user to ensure familiarity with the safe and proper use of any hazardous materials and processes, and to take necessary precautionary measures to ensure the health and safety of all personnel involved.

1.3 The values stated in SI units are to be regarded as the standard.

2. REFERENCED DOCUMENTS

2.1 SAE Publications:

Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001.

AMS 4037 Aluminum Alloy Sheet and Plate, 4.4 Cu - 1.5 Mg - 0.6 Mn (2024-T3 Flat Sheet, T351 Plate) Solution Heat Treated

2.2 ASTM Publications:

Available from ASTM, 100, Barr Harbor Drive, West Conshohocken, PA 19428-2959.

ASTM D 1193 Reagent Water

2.3 ISO Publication:

Available from International Organization for Standardization, Case Postal #56, rue Varembe, CH-1211, Switzerland.

ISO 10012: Quality assurance requirements for measuring equipment

3. SUMMARY OF TEST

3.1 This test permits the determination of laboratory anti-icing endurance times of SAE Type II, Type III, and Type IV fluids. The test fluids to be evaluated are applied to a test plate exposed to light freezing rain. Their anti-icing endurance times are evaluated by measuring the minimum exposure time before a specified degree of freezing occurs. A

general description of the anti-icing endurance time test referred to in this appendix is as follows.

3.2 Light Freezing Rain Simulation Test:

This test involves pouring the fluid onto a test plate inclined at a 10° angle from the horizontal. The anti-icing endurance time is recorded as the time for ice formation to cover 30% of the test plate. Four tests corresponding to different sets of temperature and icing intensities are produced:

Table 1: Light Freezing Rain Test Conditions

Test condition	Temperature (°C)	Icing Intensity ($g/dm^2/h$)
LZR-A	-3	13
LZR-B	-3	25
LZR-C	-10	13
LZR-D	-10	25

It is a fundamental requirement of this test that the spray impinges onto the surface of the test plate as supercooled water droplets that freeze shortly after impact. This is verified by observation of an untreated (no fluid) ice catch aluminum pan.

4. EQUIPMENT AND TEST PARAMETERS

4.1 General:

A description of the minimum requirements for the environmental test chamber and associated test equipment, including test plate, spray equipment, and data acquisition, is given below. A summary of the performance requirements for the test equipment is given in Table 2. Other spray equipment that meets the requirements of this appendix is acceptable.

4.2 Test Chamber (see Table 2):

The plate and air temperature shall be within 0.5°C prior to starting the test. The temperature sensing device shall be in proximity to the test plate, typically within 1.5 m of the side of the test plate, but outside the spray area. The distance and position of the sensing device shall be reported. The air exchange rate in the chamber shall correspond to a horizontal air velocity of no greater than 1.0 m/s when measured 50 mm above the surface of the test plate. The test chamber shall be equipped with artificial lighting such that it does not interfere with the precipitation nor with the air, fluid, and plate temperatures.

The air temperature, plate temperature, and humidity sensing devices shall be linked to an electronic data acquisition system as a means of checking the environmental control characteristics of the test chamber throughout the course of a test run.

Table 2: Summary of Performance Requirements for Test Equipment

Test Parameters	Requirements
Test Chamber	
• Distance between nozzle and plate	7 m ± 0.5
• Air temperature range	0 to -10°C
• Average set point temperature	± 0.5°C
• Standard deviation	0.3°C
• Minimum temperature sampling rate	1 datum per minute
• Horizontal air velocity	≤ 1.0 m/s
• Relative humidity	> 40%
Test Plate	
• Material	Aluminum alloy AMS 4037
• Surface finish	Average surface roughness: Ra = 0.1 to 0.2 μm
• Test panel dimensions	500 mm long x 300 mm wide x 3.2 mm thick
• Slope	10° ± 0.2
• Temperature at start of test	Within ± 0.5°C of air temperature
Ice Catch Pans	
• Ice catch pan dimensions	100 mm x 100 mm x 0.8 mm thick, with all-around rim 15 mm high
• Minimum number of reference ice catch pans per test plate	8
Spray Equipment	
• Water supply to nozzle	Conforming to ASTM D1193 Type IV
• Water supply temperature	< 2°C measured just before the nozzle
• Water droplet size	1000 μm ± 100 median volume diameter
• Water spray intensity	13 g/dm²/h ± 0.5 and 25 g/dm²/h ± 1.0

4.3 Test Plate (see Table 2):

Each test plate is a removable panel placed on a support housed within the test chamber. Each panel shall be marked with a horizontal line 150 mm from the upper shorter edge of the test plate or with any other permanent pen marking used to estimate the degree of ice formation on fluid-treated panels during the course of a test run.

Each test plate shall be equipped with a temperature sensor located on the underside of the plate. This sensor shall be capable of measuring to an accuracy of ± 0.5°C and shall be linked to an electronic data acquisition system.

The test plate support shall be set up in such a way that it can accommodate a minimum of eight 100 mm x 100 mm ice catch pans surrounding, but not in contact with, each test plate (see Figure 1). The test plate support face shall be inclined at a 10° angle ± 0.2 from horizontal. The test plates shall be placed on the support such that the fluid can freely flow off all edges of the plate. The test stand shall be designed to minimize the contact between the test surface and the support.

4.3.1 Precipitation Catch Pans:

The ice catch pans, described in Table 2 and Figure 2, are used to evaluate the ice catch, as described in section 5.4.

4.4 Spray Equipment:

4.4.1 General Requirements:

The equipment used to provide the water spray includes a low flow nozzle supplied with water conforming to ASTM D1193 Type IV. This equipment is housed in the upper region of the test chamber 7 m ± 0.5 above the test plate.

The exact type and geometry of the spray system used to generate the water spray for the test is left to the discretion of the user, provided the following parameters are met.

(a) Median volume droplet diameter of the water spray shall be 1000 μm ± 100.

(b) The average intensity of the water spray produced during a test shall correspond to light freezing rain conditions:

LZR-A and LZR-C	13 g/dm^2/h ± 0.5
LZR-B and LZR-D	25 g/dm^2/h ± 1.0

(c) The water spray shall be evenly distributed over the entire area of the test plate. Even distribution is verified by the standard deviation obtained from a calibration test described in section 5.4.

(d) To allow the droplets to become supercooled at a temperature close to that of the ambient air temperature, the distance between the nozzle and the test plate shall be of 7 m ± 0.5

To provide some background information, an example of a suitable spray system is outlined below.

4.4.2 Example of Spray Equipment:

The hydraulic nozzle shown in Figure 3 comprises three sections: an outer unit holding two inner units. A prescribed volume of ASTM D1193 Type IV water is stored in a pressurized tank and provides the flow to the nozzle. The water droplet size depends on the nozzle used, and a pulse system allows the flow of water through the nozzle to be controlled. The icing intensity is obtained by selecting the opening and closing times (on/off) of the pulse system. An even distribution over the test plate is achieved by a controlled oscillation of the nozzle.

4.5 Temperature Control Equipment:

The air temperature shall be maintained at the required level using heat exchangers connected to temperature control equipment featuring a solid state temperature sensor such as a platinum resistance probe (100 ohms at 0°C) coupled to a proportional temperature controller having a minimum resolution of 0.5°C.

4.6 Humidity Measuring Equipment:

This equipment consists of a humidity sensor of the capacitance, resistance, or conductivity type capable of covering a range of 40 to 100% RH from 0°C to -10°C.

4.7 Air Distribution System:

This system shall consist of a fan or fans to provide air recirculation through the main body of the test chamber and to the heat exchanger. Ducting for the passage of air at both the inlet and the outlet of the heat exchanger shall have entry and exit ports positioned to provide good air recirculation throughout the test chamber. The heat exchanger shall be capable of cooling the air and maintaining it at the specified temperature level. Air flow shall be measured using a suitable anemometer or velometer.

5. CALIBRATION OF TEST EQUIPMENT

5.1 Standard Measuring Devices:

All temperature sensors, humidity sensors, electronic balances, anemometers, and timing devices shall be maintained in a known state of calibration in accordance with recognized international standards such as ISO 10012, by calibrating each measuring device at least once every six months or whenever a piece of equipment is repaired, replaced, or moved, and a written record of the calibrations shall be kept available.

5.2 Average Air Velocity:

The average horizontal component of the air velocity in the test chamber, when measured 50 mm above the centerline at the upper edge of the test plate, must not exceed 1.0 m/s.

5.3 Water Droplet Size Determination:

Several methods are available to determine the water droplet sizes referred to in Table 2. The following are acceptable examples:

(a) Slide Impact Method with Oil: A sample of the water droplets from the precipitation is collected on an oil-coated microscope slide. An oil having a viscosity of about 5000 mPas at 20°C, spread to a thickness of about 2000 μm, will be suitable. The oil can be either a mineral oil or a silicone oil. The droplet size is determined by direct observation under a microscope using an eyepiece with the appropriate graticle, or from enlarged photographs of the slide.

(b) Dye-Stain Method: The dye-stain technique consists of dusting filter paper discs with a water-activated, very finely divided powder form of methylene blue dye. The prepared discs are manually positioned under precipitation for a fixed time to acquire a droplet size pattern. A calibration curve is then used to convert from the measured diameter of the droplets on the pattern to the experimental median volume diameter.

5.4 Ice Catch Calibration:

For each anti-icing endurance time test, it is important to establish that even and reproducible ice formation occurs over the surface of the test plates. To carry out this evaluation, ice catch measurements shall be performed under each condition. A summary of the test conditions follows.

Table 3: Light Freezing Rain Simulation Test Ice Catch Calibration

Condition	LZR-A	LZR-B	LZR-C	LZR-D
Air Temperature (°C)	-3	-3	-10	-10
Mean Water Spray Intensity (g/dm²/h)	13 ± 0.5	25 ± 1.0	13 ± 0.5	25 ± 1.0
Standard Deviation (g/dm²/h)	0.7	1.5	0.7	1.5
Calibration Test Duration (min)	15 and 60	15 and 30	15 and 30	15

To assess the ice catch for each condition, a total of at least 20 100 mm x 100 mm ice catch pans are used. Each test plate is replaced with 12 ice catch pans, which in turn are surrounded by at least 8 additional reference ice catch pans (see Figure 4). These preweighed pans are weighed upon completion of each test and the difference in the recorded weights is the ice catch for that pan. The average ice catch on the pans replacing the test panel is calculated as well as the average ice catch on the reference pans. It is the ratio between these two values that is used to estimate the icing intensity during a fluid test run, when only the reference pans are available, i.e:

from calibration test:

$$Ratio = \frac{I_{plate}}{I_{ref}}$$

where:

$Ratio$ = ratio of the ice catch over the pans replacing the test plate with respect to the ice catch over the reference pans

I_{plate} = the average ice catch on the pans replacing the test plate

I_{ref} = the average ice catch on the reference pans

During an anti-icing endurance time test run, the ice catch is measured on the reference pans, averaged, and the value is then is multiplied by the ratio calculated above based on a calibration test performed under the same conditions. The resulting value is the estimated icing intensity over the test panel:

for fluid test run: Estimated $I_{plate} = Ratio \times I_{ref}$

where I_{ref} is measured during a fluid test and $Ratio$ has been determined in a previous calibration test.

For a calibration test to be valid, the ice catch on the pans replacing the test plates shall fall within the variation specified in Table 3.

This calibration test shall be run at least once every six months or whenever a piece of equipment is repaired, replaced, or moved.

6. TEST PROCEDURE

6.1 Test Plate Cleanliness:

The test plates shall be free of all visible contamination, smears, or stains, except for the horizontal line marked at the 150 mm point from the upper edge of the test plate or any other marking used to estimate ice coverage. Between test runs, any contamination shall be removed by washing with hot water immediately followed by ethanol. If the same fluid is tested on the same plate for two consecutive tests, it is not necessary to clean the plates with ethanol before the second test, hot water is sufficient.

6.2 Prepare 500 mL of the candidate test fluid for each test panel to be coated. If more fluid is required, the quantity of fluid actually used shall be mentioned in the qualification statement report. The fluid shall be sheared in accordance with the specification for the respective SAE Type fluid. The test shall start within two hours after the product has been sheared, but not within the first 20 minutes after shearing.

6.3 Ensure the test chamber and fluid are at the required temperature. Verify that the test plate temperature is within 0.5°C of the air temperature. Fluids shall be applied at 20°C ± 5.

6.4 Pour the fluid onto each test plate, start the timing device, cover each test plate with a removable cover, and turn on the water spray. After five minutes, remove the cover from each test plate. Observe the panels and, when failure occurs as defined in section 6.5, record the time as the anti-icing endurance time. When the water spray is turned off, weigh the ice catch on each 100 mm x 100 mm reference ice catch pan and, using the method described in section 5.4, estimate the icing intensity for each test panel. If the icing intensity is within the specified limits for the test being conducted, the time recorded is valid for the test.

Delayed crystallization may occur during the course of a test run, and can be easily recognized by a sudden (within 30 seconds) frozen contamination coverage of a large surface of the plate. If this coverage exceeds 30% of the plate's total surface, the test is invalid and must be repeated. In the case of suspected delayed crystallization, if the test has been invalidated 3 times, the plate may be seeded at its center top edge with an ice crystal to initiate crystallization. Seeding consists of putting an ice crystal in contact with the fluid by means of a chilled metal rod (below 0°C). If the ice suddenly covers more than 30% of the seeded plate's surface, the test is invalid.

6.5 Failure Criterion:

Failure is called when 30% of the plate is covered with frozen contamination.
Examples of the appearance of this frozen contamination include, but are not limited to:

- Ice front
- Ice sheet
- Slush, in clusters or as a front
- Disseminated fine ice crystals
- Frost on surface
- Clear ice pieces partially or totally imbedded in fluid

Pen marks on the plate can be used to estimate the area of failure. For instance, a line drawn across the plate at 150 mm from the top edge will delineate an area corresponding to 30% of the plate.

6.6 Reproducibility/Precision:

The light freezing rain simulation test is dynamic by nature, and small variations can be expected. Each test condition shall be tested on at least two panels. If the maximum variation of the anti-icing endurance time is less than 10% of the average anti-icing endurance time, report this time. If not, repeat testing on two additional panels, for a total of four data points. The highest and lowest points shall be discarded, and the average of the two remaining values shall be the anti-icing endurance time for that condition.

6.7 Report:

The report shall state the name and address of the facility conducting the tests, together with a statement confirming the test facility is autonomous of the manufacturer or vendor of the fluid. The following information shall also appear on the test document:

Date tests conducted
Manufacturer or vendor's name and address
Name or reference number and lot number of the fluid tested
Type of fluid (SAE Type II, SAE Type III, or SAE Type IV)
Summary of the test results, and ice catch for each test
Method of estimation of failure area
Description of appearance of frozen contamination (section 6.5)

Condition of fluid: Concentrate as supplied to test facility, or diluted with hard water as defined in the specification and subsequently sheared in accordance with specification giving the dilution as a ratio (example: SAE Type II diluted 75/25 with hard water).

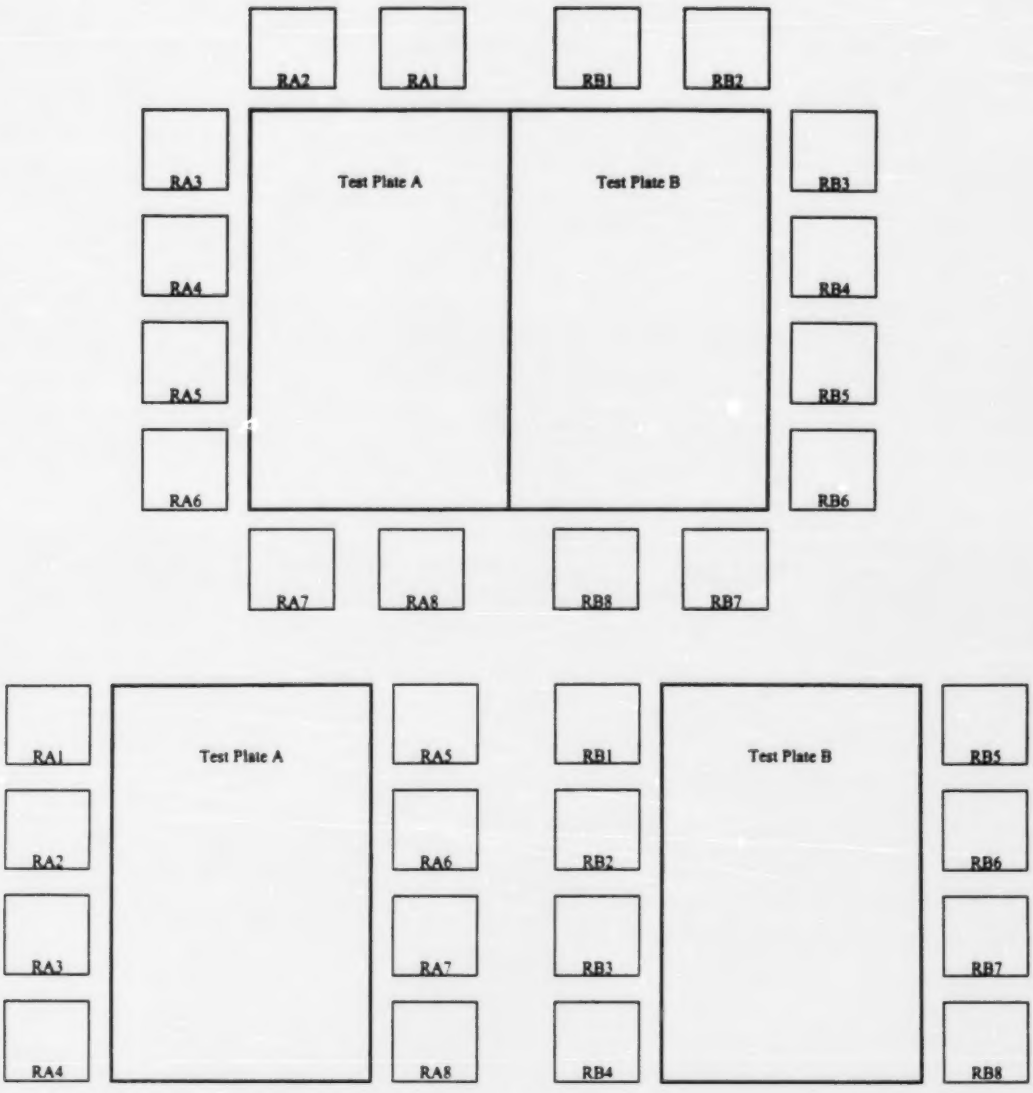


FIGURE 1 – Two examples where two test plates are each surrounded by 8 reference ice catch pans, used for the ice catch estimation

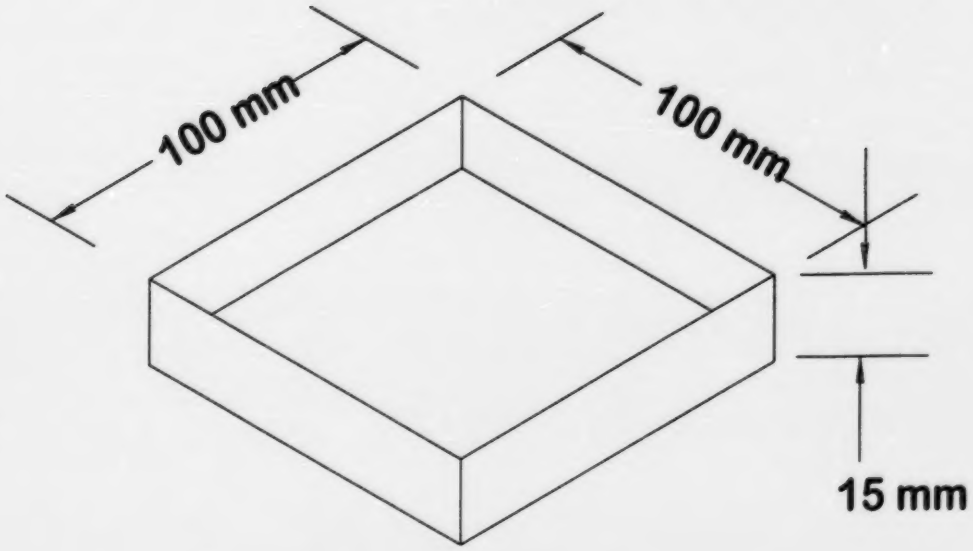
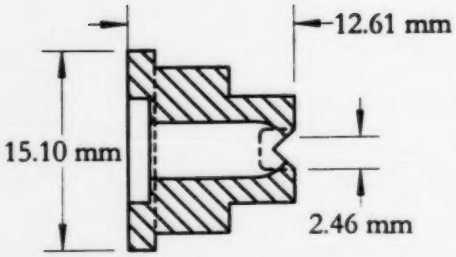
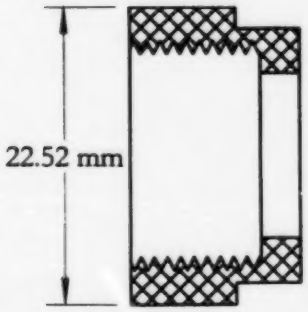


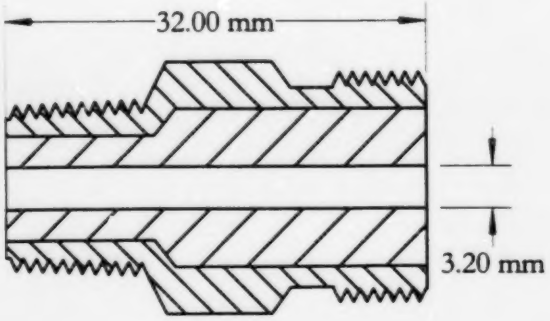
FIGURE 2 – Ice catch pan



Piece A



Piece B



Piece C

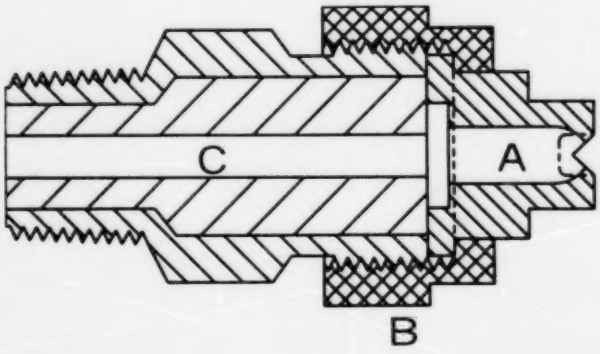


FIGURE 3 – Example of a hydraulic nozzle for light freezing rain

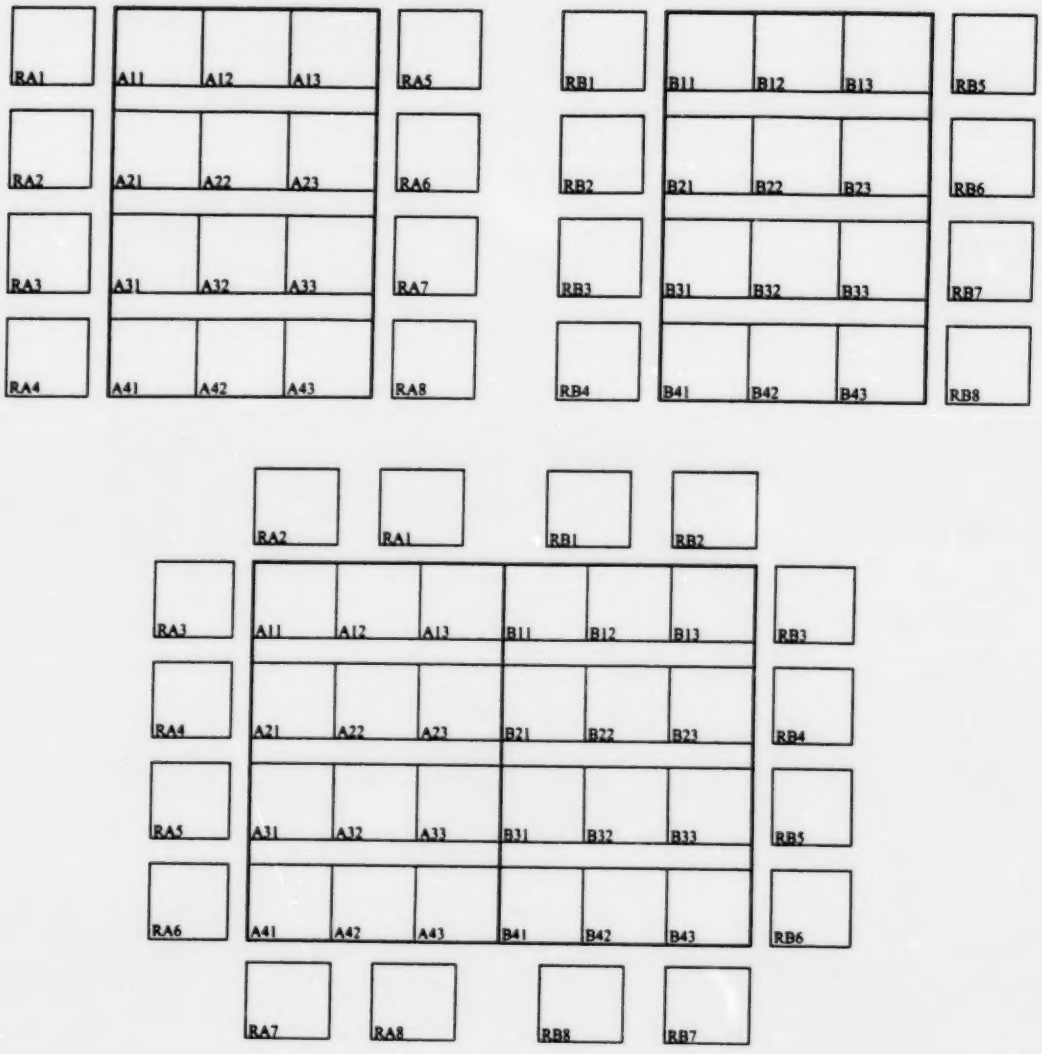


FIGURE 4 – Two examples of dispositions of reference ice catch pans (denoted RA1 to RA8 for plate A and RB1 to RB8 for plate B), each surrounding the 12 ice catch pans replacing the test plates (A11 to A43 for test plate A and B11 to B43 for test plate B)

APPENDIX FRST

PROCEDURES FOR ANTI-ICING ENDURANCE TIME TESTING OF AIRCRAFT DEICING/ANTI-ICING FLUID UNDER FROST CONDITIONS

1. SCOPE

1.1 This document establishes the minimum requirements for an environmental test chamber and the test procedures used to carry out anti-icing endurance time tests of aircraft deicing/anti-icing fluids under frost conditions. The primary purpose for such a test method is to determine the frost anti-icing endurance time under controlled laboratory conditions for SAE Type II, Type III, and Type IV fluids.

1.2 While the materials, methods, applications, and processes described or referenced in this appendix may involve the use of hazardous materials, this appendix does not address the hazards that may be involved in such use. It is the sole responsibility of the user to ensure familiarity with the safe and proper use of any hazardous materials and processes, and to take necessary precautionary measures to ensure the health and safety of all personnel involved.

1.3 The values stated in SI units are to be regarded as the standard.

2. REFERENCED DOCUMENTS

2.1 SAE Publications:

Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001.

AMS 4037 Aluminum Alloy Sheet and Plate, 4.4 Cu - 1.5 Mg - 0.6 Mn (2024-T3 Flat Sheet, T351 Plate) Solution Heat Treated

2.2 ASTM Publications:

Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

ASTM D 1193 Reagent Water

2.3 ISO Publication:

Available from International Organization for Standardization, Case Postal #56, rue Varembe, CH-1211, Switzerland.

ISO 10012: Quality assurance requirements for measuring equipment

3. SUMMARY OF TEST

3.1 This test permits the determination of laboratory anti-icing endurance times of SAE Type II, Type III, and Type IV fluids. The test fluids to be evaluated are applied to a test plate exposed to frost. Their anti-icing endurance times are evaluated by measuring the

minimum exposure time before a specified degree of freezing occurs. A general description of the anti-icing endurance time test referred to in this appendix is as follows.

3.2 Frost Simulation Test:

This test involves pouring the fluid onto a test plate inclined at a 10° angle from the horizontal. The test plate is refrigerated to 3°C below the temperature of the air and exposed to a high humidity environment. The anti-icing endurance time is recorded as the time for ice formation to cover 50% of the test plate. Five tests corresponding to different sets of temperature and icing intensity conditions are produced:

Table 1: Frost Test Conditions

Test Condition	Air Temperature (°C)	Icing Intensity ($g/dm^2/h$)
Frst-A	0	0.20
Frst-B	-3	0.20
Frst-C	-14	0.13
Frst-D	-25	0.05
Frst-E	<-25*	report**

** This test will be performed at the lowest usable temperature of the fluid if it is below -25°C.*

*** These values will depend on the actual air and plate temperature.*

It is a fundamental requirement of this test that the Relative Humidity is maintained elevated without any visible precipitation (such as mist, fog, or drizzle).

4. EQUIPMENT AND TEST PARAMETERS

4.1 General:

A description of the minimum requirements for the environmental test chamber and associated test equipment, including test plate chilling unit, test plate, humidity generator, and data acquisition, is given below. A summary of the performance requirements for the test equipment is given in Table 2. Other humidity control equipment that meets the requirements of this appendix is acceptable.

4.2 Test Chamber (see Table 2):

The temperature sensing device shall be in proximity to the test plate, typically within 1.5 m of the side of the test plate. The distance and position of the sensing device shall be reported. The air exchange rate in the chamber shall correspond to an average horizontal air velocity of 0.5 m/s ± 0.1 when measured 50 mm above the surface of the test plate. The test chamber shall be equipped with artificial lighting such that it does not interfere with air fluid and plate temperatures.

The humidity can be produced using a saturated water vapor generator housed in the exit side of the air recirculating system, and controlled using a suitably calibrated humidity sensor linked to a control system. The humidity sensor shall be placed 50 mm above the surface of the test plate at the center line of the upper edge of the test plate and within 300 mm behind. Both the air temperature and humidity sensing devices shall be linked to an electronic data acquisition system as a means of checking the environmental characteristics of the test chamber throughout the course of a test run.

Table 2: Summary of Performance Requirements for Test Equipment

Test Parameters	Requirements
Test Chamber	
• Minimum volume	1 m^3 for each 2.25 dm^2 test panel surface
• Air temperature range	0 to at least -25°C
• Average set point temperature	± 0.5 at 0, -3 and -14°C and ± 2 at -25°C and below -25°C
• Standard deviation	± 0.2 at 0, -3 and -14°C and ± 0.5 at -25°C and below -25°C
• Minimum temperature sampling rate	1 datum per minute
• Horizontal air velocity	0.5 m/s ± 0.1
• Relative Humidity	> 94% at -0°C > 90% at -3°C > 80% at -14°C > 70% at -25°C
Test Plate	
• Material	Aluminum alloy AMS 4037
• Surface finish	Average surface roughness: Ra = 0.1 µm to 0.2
• Test panel dimensions	300 mm long x 100 mm wide x 1.6 mm thick
•	
• Slope	10° ± 0.2
• Temperature range	0 to at least -28°C capable of a temperature 3°C below the air temperature
Ice Catch Plates	
• Ice catch plate dimensions	100 mm x 100 mm x 1.6 mm thick
• Number of ice catch plates per test plate	3

4.3 Test Plate Cooling:

The test plate is a removable 100 x 300 mm panel placed on the face of the chiller unit. Both the test plate and the chiller unit are housed within the test chamber.

The chiller unit face shall be inclined at a 10° angle ± 0.2 from the horizontal. The upper surface of the chiller unit face shall comprise at least six panels, each separated by a divider that will protrude 5 mm above the surface of the test plates to prevent the possibility of cross-contamination between fluid applied to adjacent panels. Each panel shall be marked with a horizontal line running across and 150 mm from the upper shorter edge of the test plate or with any other permanent pen marking used to estimate the degree of ice formation on fluid-treated panels during the course of a test run.

The lower surface of the chiller unit face shall be coupled to a fluid cell capable of accepting a recirculating supply of heat transfer fluid such that the upper surface of the test plate can be maintained at a temperature 3°C ± 0.5 below the air temperature. The temperature sensing device shall be mounted in the upper surface of the chiller unit as close as possible to the test surface. This temperature sensor shall be linked to an electronic data acquisition system to check and record the test plate temperature throughout the course of a test run.

4.3.1 Precipitation Catch Plates:

The ice catch plates described in Table 2 are used to evaluate the ice catch, as described in section 5.4.

4.4 Humidity Control Equipment:

Relative humidity shall be maintained at the specified level using a saturated water vapor generator and a humidity sensor of the capacitance, resistance or conductivity type capable of covering a range of 70 to 100% relative humidity.

4.5 Temperature Control Equipment:

Both the air and test plate temperatures shall be maintained at the required level using heat exchangers connected to temperature control equipment featuring a solid state temperature sensor such as a platinum resistance probe (100 ohms at 0°C) coupled to a proportional temperature controller having a minimum resolution of 0.5°C.

4.6 Air Distribution System:

This system shall consist of a fan or fans to provide air recirculation through the main body of the test chamber and to the heat exchanger. Ducting for the passage of air at both the inlet and the outlet of the heat exchanger shall have entry and exit ports positioned to provide good air recirculation throughout the test chamber. The heat exchanger shall be capable of cooling the air and maintaining it at the specified temperature level. Air flow shall be measured using a suitable anemometer or velometer.

5. CALIBRATION OF TEST EQUIPMENT

5.1 Standard Measuring Devices:

All temperature sensors, humidity sensors, electronic balances, anemometers, and timing devices shall be maintained in a known state of calibration in accordance with recognized international standards such as ISO 10012, by calibrating each instrument at least once every six months or whenever a piece of equipment is repaired, replaced, or moved, and a written record of the calibrations shall be kept available.

5.2 Average Air Velocity:

The average horizontal component of the air velocity in the test chamber, when measured 50 mm above the centerline at the upper edge of the test plate, must be 0.5 m/s ± 0.1.

5.3 Ice Catch Calibration:

For each anti-icing endurance time test, it is important to establish that even and reproducible ice formation occurs over the surface of the test plates. To carry out this evaluation, ice catch measurements shall be performed under each condition. A summary of the test conditions follows.

To assess the ice catch for each condition, a total of eighteen 100 mm x 100 mm ice catch plates are used. Each 300 x 100 mm test panel is replaced with three 100 x 100 mm test plates. These preweighed panels are weighed upon completion of each test and the difference in the recorded weights is the ice catch for that plate. The average ice catch over the test surface shall correspond to frost accumulation for that test condition and variation shall be within the limits specified in Table 3.

During the course of a test run, each test plate shall be replaced with three (3) 100 x 100 mm ice catch plates. A calibration in which all test plates are replaced by ice

catch plates shall be carried out at least once every six months or whenever a piece of equipment is repaired, replaced, or moved.

Table 3: Frost Ice Catch Calibration for Each Test Condition

Condition	FRST-A	FRST-B	FRST-C	FRST-D	FRST-E
Air Temperature (°C)	0 ± 0.5	-3 ± 0.5	-14 ± 0.5	-25 ± 1.0	< -25 ± 1*
Plate Temperature (°C)	-3 ± 0.5	-6 ± 0.5	-17 ± 0.5	-28 ± 1.0	3°C below air
Relative Humidity	> 94%	> 90%	> 80%	> 70%	report**
Frost Accumulation ($g/dm^2/h$)	0.20 ± 0.02	0.20 ± 0.02	0.13 ± 0.01	0.05 ± 0.01	report**
Maximum Variation ($g/dm^2/h$)	± 0.03	± 0.03	± 0.02	± 0.01	report**
Calibration Test Duration (h)	12 and 3	12 and 3	12 and 3	12 and 4	12 and 4

**These values will depend on the actual air and plate temperatures.*

*** This test will be performed at the lowest usable temperature of the fluid if it is below -25°C.*

6. TEST PROCEDURE

6.1 Test Plate Cleanliness:

The test plates shall be free of all visible contamination, smears, or stains, except for the horizontal line marked at the 150 mm point from the upper edge of the test plate or any other marking used to estimate ice coverage. Between test runs, any contamination shall be removed by washing with hot water immediately followed by ethanol . If the same fluid is tested on the same plate for two consecutive tests, it is not necessary to clean the plates with ethanol before the second test, hot water is sufficient.

6.2 Prepare 500 mL of the candidate test fluid. The fluid shall be sheared in accordance with the specification for the respective SAE Type fluid. The test shall start within two hours after the fluid has been sheared but not within the first 20 minutes after shearing. Each test panel shall be coated with 115 mL or 120 g of fluid. If more fluid is required, the quantity of fluid actually used shall be mentioned in the report qualification statement.

6.3 Place three test panels in alternation with three sets of ice catch plates on the chiller unit (see Figure 4). Protect all plates with paper towels. Ensure the test chamber and test plate support are at the required temperature. Fluids shall be applied at 20°C ± 0.5.

6.4 Pour the fluid onto each test plate, start the timing device, and cover the entire test surface with a removable cover. Turn on the humidity generator (the humidity generator may be turned on before setting the panels and the plates as long as the upper surface of the chiller unit is adequately protected). After 5 minutes, remove the cover from the test surface. Observe the panels and, when failure occurs as defined in section 6.5, record the time of this event. When the humidity generator is turned off, weigh the ice catch on each 100 mm x 100 mm section of the uncoated panels and determine the icing intensity. If the icing intensity is within the specified limits for the test being conducted, the time is valid for the frost simulation test.

Delayed crystallization may occur during the course of a test run, and can be easily recognized by a sudden (within 30 seconds) coverage of a large surface of the plate with frozen contamination. If this coverage exceeds 50% of the plate's total surface, the test is invalid and must be repeated. In the case of suspected delayed crystallization, if the test has been invalidated 3 times, the plate may be seeded at its center top edge with an ice crystal to initiate crystallization. Seeding consists of putting an ice crystal in contact with

the fluid by means of a chilled metal rod (below 0°C). If the ice covers more than 50% of the seeded plate's surface, the test is invalid.

6.5 Failure criterion:

Failure is called when 50% of the plate is covered with frozen contamination. Examples of the appearance of this frozen contamination include, but are not limited to:

- Ice front
- Ice sheet
- Slush, in clusters or as a front
- Disseminated fine ice crystals
- Frost on surface
- Clear ice pieces partially or totally imbedded in fluid

Pen marks on the plate can be used to estimate the area of failure. For instance, a line drawn across the plate at 150 mm from the top edge will delineate an area corresponding to 50% of the plate.

6.6 Reproducibility/Precision:

The frost simulation test is dynamic by nature, and small variations can be expected. Each test condition shall be tested on at least three panels. If the maximum variation of the anti-icing endurance time is less than 10% of the average anti-icing endurance time, report this time. If not, repeat testing on three additional panels, for a total of six data points. The highest and lowest points should be discarded and the average of the four remaining values shall be the anti-icing endurance time for that condition.

6.7 Report:

The report shall state the name and address of the facility conducting the tests, together with a statement confirming the test facility is autonomous of the manufacturer or vendor of the fluid. The following information shall also appear on the test document:

Date tests conducted
Manufacturer or vendor's name and address
Name or reference number and lot number of the fluid tested
Type of fluid (SAE Type II, SAE Type III, or SAE Type IV)
Summary of test results, and the ice catch results for each test
Method of estimation of failure area
Description of appearance of frozen contamination (section 6.5)

Condition of fluid: Concentrate as supplied to test facility, or diluted with hard water as defined in the specification and subsequently sheared in accordance with specification giving the dilution as a ratio (example: SAE Type II diluted 75/25 with water).

Table 4: Frost Simulation Tests – Proposed Order of Fluid Application to a Chiller Unit Having Six Panels

	Panel #	1	2	3	4	5	6
Run #	1	BLANK (A)	FLUID	BLANK	FLUID	BLANK	FLUID
	2 (B)	FLUID	BLANK	FLUID	BLANK	FLUID	BLANK

(A) BLANK panels act as controls to measure the ice catch after each run.
(B) Run #2 only if required.

APPENDIX CSW

PROCEDURES FOR ANTI-ICING ENDURANCE TIME TESTING OF AIRCRAFT DE-ICING/ANTI-ICING FLUID UNDER RAIN ON A COLD-SOAKED WING CONDITIONS

1. SCOPE

1.1 This document establishes the minimum requirements for an environmental test chamber and the test procedures to carry out anti-icing endurance time tests of aircraft deicing/anti-icing fluids under the condition of rain on a cold-soaked wing. The primary purpose for such a test method is to determine the rain on a cold-soaked wing anti-icing endurance time under controlled laboratory conditions for SAE Type II, Type III, and Type IV fluids.

1.2 While the materials, methods, applications, and processes described or referenced in this appendix may involve the use of hazardous materials, this appendix does not address the hazards that may be involved in such use. It is the sole responsibility of the user to ensure familiarity with the safe and proper use of any hazardous materials and processes, and to take necessary precautionary measures to ensure the health and safety of all personnel involved.

1.3 The values stated in SI units are to be regarded as the standard.

2. REFERENCED DOCUMENTS

2.1 SAE Publications:

Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001.

AMS 4037 Aluminum Alloy Sheet and Plate, 4.4 Cu - 1.5 Mg - 0.6 Mn (2024-T3 Flat Sheet, T351 Plate) Solution Heat Treated

2.2 ASTM Publications:

Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

ASTM D 1193 Reagent Water

2.3 ISO Publication:

Available from International Organization for Standardization, Case Postal #56, rue Varembe, CH-1211, Switzerland.

ISO 10012: Quality assurance requirements for measuring equipment

2.4 Other Publications:

Available from the Transportion Development Centre, 800 René Lévesque Blvd. W., Suite 600, Montreal, QC H3B 1X9

Validation of Methodology for Simulating a Cold Soaked Wing, TP 12899E.

3. SUMMARY OF TEST

3.1 This test permits the determination of laboratory rain on a cold-soaked wing anti-icing endurance times of SAE Type II, Type III, and Type IV fluids. The fluids to be evaluated are applied to a test plate cooled to -10°C and exposed to rain and drizzle at +1°C. Their anti-icing endurance times are evaluated by measuring the minimum exposure time before a specified degree of freezing occurs. A general description of the anti-icing endurance time tests referred to in this appendix is as follows.

3.2 Rain on a Cold-Soaked Wing Simulation Test:

This test involves pouring the fluid onto a test plate inclined at a 10° angle from horizontal, cold soaked to -10°C in a climatic chamber refrigerated to 1°C. The anti-icing endurance time is recorded as the time for ice formation to cover 30% of the test plate. Two conditions of precipitation conditions are produced:

Table 1: Rain on a Cold-Soaked Wing Test Conditions

Test Condition	Air Temperature (°C)	Precipitation Rate ($g/dm^2/h$)
RCSW-1	1	5
RCSW-2	1	75

4. EQUIPMENT AND TEST PARAMETERS

4.1 General:

A description of the minimum requirements for the environmental test chamber and associated test equipment, including cold soak box, test plate, spray equipment, and data acquisition, is given below. A summary of the performance requirements for the test equipment is given in Table 2. Other spray equipment that meets the requirements of this appendix is acceptable.

4.2 Test Chamber (see Table 2):

The test chamber used to perform rain on a cold-soaked wing simulation tests shall be capable of air temperature control in the range 0 to 2°C with an accuracy of ± 0.5°C. The temperature sensing device shall be in proximity with the test plate, typically within 1.5 m of the side of the test plate, but outside the spray area. The distance and position of the sensing device shall be reported. The air exchange rate in the chamber shall correspond to a horizontal air velocity no greater than 1.0 m/s when measured 50 mm above the surface of the test plate. The test chamber shall be equipped with artificial lighting such that it does not interfere with the precipitation nor with the air, fluid, and plate temperatures.

The air temperature, plate temperature, and humidity sensing devices shall be linked to an electronic data acquisition system as a means of checking the environmental control characteristics of the test chamber throughout the course of a test run.

Table 2: Summary of Performance Requirements for Test Equipment

Test Parameters	Requirements
Test Chamber	
• Distance between nozzle and test plate	7 m ± 0.5
• Air temperature	
• Average set point temperature	1°C
• Standard deviation	± 0.5°C
• Minimum temperature sampling rate	0.3°C
• Horizontal air velocity	1 datum per minute
• Relative humidity	≤ 1.0 m/s
	> 40%
Test Plate	
• Material	Aluminum alloy AMS4037
• Surface finish	Average surface roughness: Ra = 0.1 to 0.2 µm
• Test panel dimensions	500 mm long x 300 mm wide x 3.2 mm thick
• Slope	10° ± 0.2 from horizontal
Precipitation catch pans	
• Precipitation catch pan dimensions	100 mm x 100 mm x 0.8 mm thick, with all-around rim 15 mm high
• Minimum number of reference precipitation catch plates	8
Cold soak box	
• Material	Aluminum alloy AMS4037, 1.6 mm thick
• Dimensions	430 mm x 300 mm x 75 mm
• Plate surface temperature at start of test	-10°C ± 1.0
• Coolant in box	65% propylene glycol, 35% water
• Coolant temperature before starting test	-17°C ± 1
Spray Equipment	
• Water supply to nozzle	Conforming to ASTM D1193 Type IV
• Water supply temperature	< 2°C measured just before the nozzle
• Water spray intensity:	
Condition: RCSW-1	5 g/dm²/h ± 0.2
RCSW-2	75 g/dm²/h ± 3.0
• Water droplet size:	
Condition: RCSW-1	250 µm ± 50 median volume diameter
RCSW-2	1400 µm ± 150 median volume diameter

4.3 Test Plate (See Table 2):

Each test area is either the upper surface of the cold soak box or a removable panel centered on the face of the cold soak box (Figure 1). If a removable panel is used, a 65/35 propylene glycol/water solution is spread between the cold soak box and the test panel to ensure that the test panel has the same temperature as the cold soak box surface. Each panel shall be marked with a horizontal line 150 mm from the upper shorter edge of the test plate or with any other permanent pen marking used to estimate the degree of ice formation on the fluid-treated panel during the course of a test run. During the course of a

test run, each test plate is surrounded by at least eight 100 x 100 mm precipitation catch pans. Both the test plate and the cold soak box are housed within the test chamber.
The cold-soak box shall be filled with propylene glycol diluted 65/35 with water at a temperature of -17°C at the start of the test. The box shall be contained within a 25 mm polystyrene insulating jacket (RSI = 1.3) during the test. The box shall be equipped with a temperature sensing device capable of measuring the temperature of the test plate with an accuracy of ± 0.5°C, and situated 150 mm from the top and 150 mm from the side of the test plate. This temperature sensor shall be linked to a data acquisition system to check and record the test plate temperature throughout the course of a test run. The cold soak box face shall be inclined at a 10° angle ± 0.2 from horizontal. The test plate is positioned on the box so that fluid can freely flow off all edges of the plate.

4.3.1 Precipitation Catch Pans:

The precipitation catch pans described in Table 2 and Figure 2, are used to evaluate the precipitation catch, as described in section 5.4.

4.4 Spray Equipment:

4.4.1 General Requirements:

The equipment used to provide the water spray includes a low flow nozzle supplied with water conforming to ASTM D1193 Type IV. This equipment is housed in the upper region of the test chamber 7 m ± 0.5 above the test plate.

The exact type and geometry of the spray system used to generate the water spray for the test is left to the discretion of the user, provided the following parameters are met.

(a) Median volume droplet diameter of the water spray shall be for condition:
RCSW-1: 250 ± 50 µm median volume diameter
RCSW-2: 1400 ± 150 µm median volume diameter

(b) The average intensity of the water spray produced during a test shall correspond to rain on a cold-soaked wing condition:
RCSW-1: 5 g/dm² ± 0.2 per hour
RCSW-2: 75 g/dm² ± 3.0 per hour

(c) The water spray shall be evenly distributed over the entire area of the test plate. Even distribution is verified by the standard deviation obtained from a calibration test described in section 5.4.

To provide some background information, an example of a suitable spray system is outlined below.

4.4.2 Example of Spray Equipment:

The hydraulic nozzles shown in Figures 3 and 4 comprise three sections: an outer unit holding two inner units. A prescribed volume of ASTM D1193 Type IV water is stored in a pressurized tank and provides the flow to the nozzle. The water droplet size depends on the nozzle used, and a pulse system allows the flow of the water through the nozzle to be controlled. The precipitation intensity is obtained by selecting the opening and closing times (on/off) of the pulse system. An even distribution over the test plate is achieved by a controlled oscillation of the nozzle.

4.5 Temperature Control Equipment:

The air temperature shall be maintained at the required level using heat exchangers connected to temperature control equipment featuring a solid state temperature sensor such as a platinum resistance probe (100 ohms at 0°C) coupled to a proportional temperature controller having a minimum resolution of 0.5°C.

4.6 Humidity Measurement Equipment:

Humidity measurement equipment consists of humidity sensors of the capacitance, resistance, or conductivity type, capable of covering a range of 40 to 100% RH at 1°C.

4.7 Air Distribution System:

This system shall consist of a fan or fans to provide air recirculation through the main body of the test chamber and to the heat exchanger. Ducting for the passage of air at both the inlet and the outlet of the heat exchanger shall have entry and exit ports positioned to provide good air recirculation throughout the test chamber. The heat exchanger shall be capable of cooling the air and maintaining it at the specified temperature level. Air flow shall be measured using a suitable anemometer or velometer.

5. CALIBRATION OF TEST EQUIPMENT

5.1 Standard Measuring Devices:

All temperature sensors, humidity sensors, electronic balances, anemometers, and timing device shall be maintained in a known state of calibration in accordance with recognized international standards such as ISO 10012, by calibrating each device at least once every six months or whenever a piece of equipment is repaired, replaced, or moved, and a written record of all calibrations shall be kept.

5.2 Average Air Velocity:

The average horizontal component of the air velocity in the test chamber, when measured 50 mm above the centerline at the upper edge of the test plate, must not exceed 1.0 m/s.

5.3 Water Droplet Size Determination:

Several methods are available to determine the water droplet sizes referred to in Table 2. The following are acceptable examples:

(a) Slide Impact Method with Oil: A sample of the water droplets from the precipitation is collected on an oil-coated microscope slide. An oil having a viscosity of about 5000 mPas at 20°C, spread to a thickness of about 1000 µm to measure the 275 µm MVD droplets and 2000 µm droplets, will be suitable. The oil can be either a mineral oil or a silicone oil. The droplet size is determined by direct observation under a microscope using an eyepiece with the appropriate graticle, or from enlarged photographs of the slide.

(b) Dye-Stain Method: The dye-stain technique consists of dusting filter paper discs with a water-activated, very finely divided powder form of methylene blue dye. The prepared discs are manually positioned under precipitation for a fixed time to acquire a droplet size pattern. A calibration curve is then used to convert from the measured diameter of the droplets on the pattern to the experimental median volume diameter.

5.4 Precipitation Catch Calibration:

It is important to establish that even and reproducible precipitation occurs over the surface of the test plates. To carry out this evaluation, precipitation catch measurements shall be performed under the appropriate test conditions. A summary of the test conditions follows.

Table 3: Rain on a Cold-Soaked Wing Simulation

Condition	CSW-1	CSW-2
Air Temperature (°C)	1 ± 0.5°C	1 ± 0.5°C
Test Plate Temperature at the Beginning of the Test	-10°C ± 1	-10°C ± 1
Coolant Temperature	-17°C ± 1	-17°C ± 1
Mean Water Spray Intensity (g/dm²/h)	5.0 ± 0.2	75.0 ± 3.0
Standard Deviation (g/dm²/h)	0.3	4.5
Calibration Test Duration (min)	15 and 60	15

To assess the precipitation catch for each condition, a total of at least 20 100 x 100 mm ice catch pans are used. Each test plate is replaced with at least 12 precipitation catch pans, which in turn are surrounded by at least 8 additional reference precipitation catch pans (see Figure 5). These preweighed pans are weighed upon completion of each test and the difference in the recorded weights is the precipitation catch for that pan. The average precipitation catch on the pans replacing the test plate is calculated, as well as the average precipitation catch on the reference pans. It is the ratio between these two values that is used to estimate the icing intensity during a fluid test run when only the reference pans are available, i.e:

from calibration test:

$$Ratio = \frac{I_{plate}}{I_{ref}}$$

where:

$Ratio$ = ratio of the precipitation catch over the pans replacing the test plate with respect to the precipitation catch on the reference pans

I_{plate} = the average precipitation catch on the pans replacing the test plate

I_{ref} = the average precipitation catch on the reference pans

During an anti-icing endurance time test run, the precipitation catch is measured on the reference pans, averaged, and this value is then multiplied by the ratio calculated above based on calibration test performed under the same conditions. The resulting value is the estimated precipitation intensity over the test plate:

for fluid test run: Estimated $I_{plate} = Ratio \times I_{ref}$

where I_{ref} is measured during a fluid test and $Ratio$ has been determined in a previous calibration test.

For a calibration test to be valid, the precipitation catch on the pans replacing the test plates shall fall within the variation specified in Table 3.

This calibration test shall be run at least once every six months or whenever a piece of equipment is repaired, replaced, or moved.

6. TEST PROCEDURE

6.1 Test Plate Cleanliness:

Prior to the refrigeration of the plate, the test plates shall be free of all visible contamination, smears, or stains, except for the horizontal line marked at the 150 mm point from the upper edge of the test plate or any other marking used to estimate precipitation coverage. Between test runs, any contamination shall be removed by washing with hot water immediately followed by ethanol. If the same fluid is tested on the same plate for two consecutive tests, it is not necessary to clean the plates with ethanol before the second test, hot water is sufficient.

6.2 The cold soak box shall be filled with a 65/35 propylene glycol/water mixture and refrigerated to -17°C.

6.3 Prepare 500 mL of the candidate test fluid for each test panel to be coated. If more fluid is required, the quantity of fluid actually used shall be mentioned in the report qualification statement. The fluid shall be sheared in accordance with the specification for the respective SAE Type fluid. The test shall start within 2 hours after the product has been sheared, but not within the first 20 minutes after shearing. Fluids shall be applied at 20°C ± 5.

6.4 Ensure the test chamber and cold soak box are at the required temperatures. If a removable test plate is used, spread the 65/35 propylene glycol/water mixture over the cold soak box before placing the test plate on the box.

6.5 Cover each test plate with paper towel as well as the entire test surface with a removable cover. When the temperature sensor within the test plate reaches -10°C, remove the cover from each test plate, apply the test fluid, and replace the removable cover. Turn on the water spray, wait five minutes, and remove the cover. Observe the test plates and, when failure occurs as defined in section 6.5, record this time as the anti-icing endurance time. When the water spray is turned off, weigh the precipitation catch on each 100 mm x 100 mm reference precipitation catch pan. Using the calibration described in section 5.4, estimate the precipitation intensity for each test plate. If the precipitation intensity is within the specified limits for the test being conducted, the time recorded is valid for that test.

Delayed crystallization may occur during the course of a test run, and can be easily recognized by a sudden (within 30 seconds) frozen contamination coverage of a large surface of the plate. If this coverage exceeds 30% of the plate's total surface, the test is invalid and must be repeated. In the case of suspected delayed crystallization, if the test has been invalidated 3 times, the plate may be seeded at its center top edge with an ice crystal to initiate crystallization. Seeding consists of putting an ice crystal in contact with the fluid by means of a chilled metal rod (below 0°C). If the ice suddenly covers more than 30% of the seeded plate's surface, the test is invalid.

6.6 Failure criterion:

Failure is called when 30% of the plate is covered with frozen contamination. Examples of the appearance of this frozen contamination include, but are not limited to:

- Ice front
- Ice sheet
- Slush, in clusters or as a front
- Disseminated fine ice crystals
- Frost on surface
- Clear ice pieces partially or totally imbedded in fluid

Pen marks on the plate can be used to estimate the area of failure. For instance, a line drawn across the plate at 150 mm from the top edge will delineate an area corresponding to 30% of the plate.

6.7 Reproducibility/Precision:

The cold-soaked wing test is dynamic by nature, and small variations can be expected. Each test condition shall be tested on at least two panels. If the maximum variation of the anti-icing endurance time is less than 10% of the average anti-icing endurance time, report this time. If not, repeat testing on two additional panels, for a total of four data points. The highest and lowest points shall be discarded, and the average of the two remaining values shall be the anti-icing endurance time for that condition.

6.8 Report:

The report shall state the name and address of the facility conducting the tests, together with a statement confirming the test facility is autonomous of the manufacturer or vendor of the fluid. The following information shall also appear on the test document:

Date tests conducted
Manufacturer or vendor's name and address
Name or reference number and lot number of the fluid tested
Type of fluid (SAE Type II, SAE Type III, or SAE Type IV)
Summary of test results, and the ice catch results for each test
Method of estimation of failure area
Description of appearance of frozen contamination (section 6.6)

Condition of fluid: Concentrate as supplied to test facility, or diluted with hard water as defined in the specification and subsequently sheared in accordance with specification giving the dilution as a ratio (example: SAE Type II diluted 75:25 with hard water).

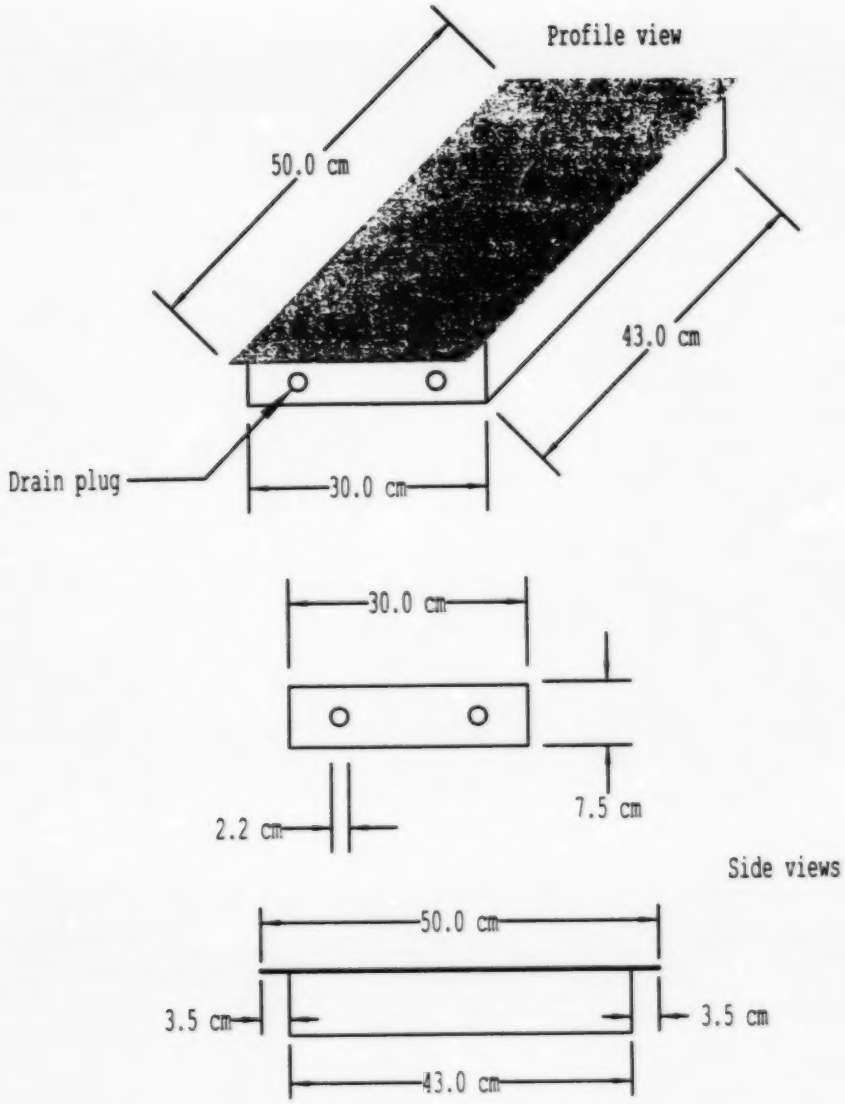


FIGURE 1 – Cold soak box and test plate

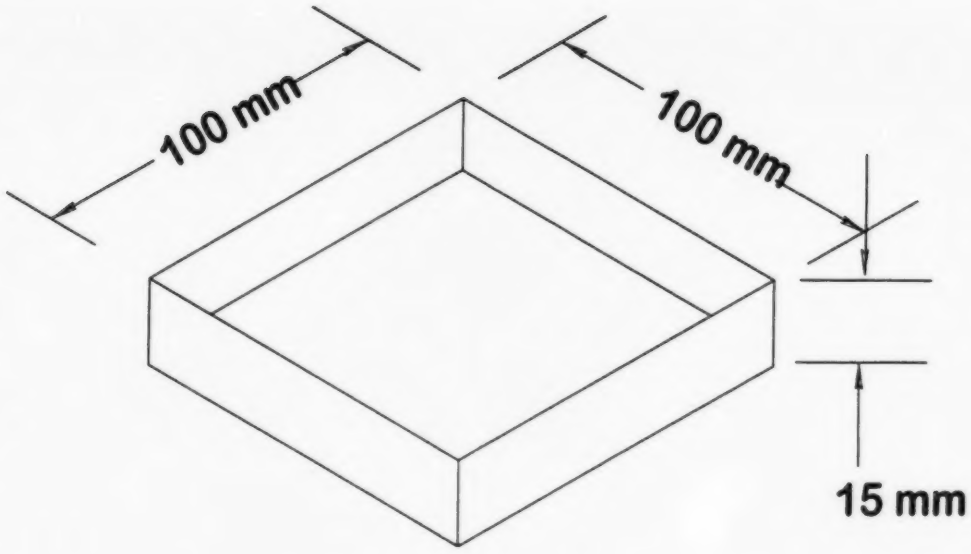


FIGURE 2 – Precipitation catch pan

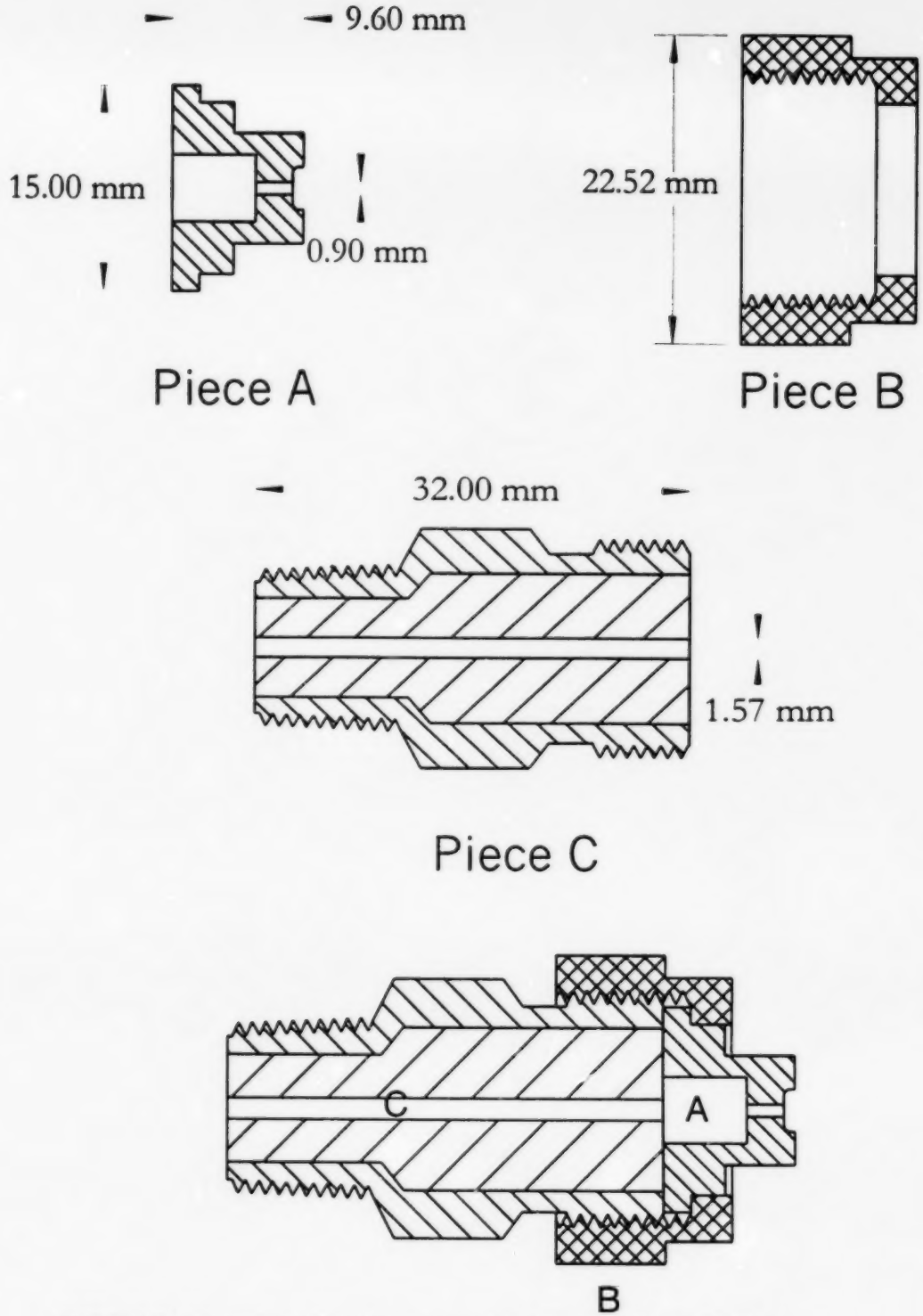
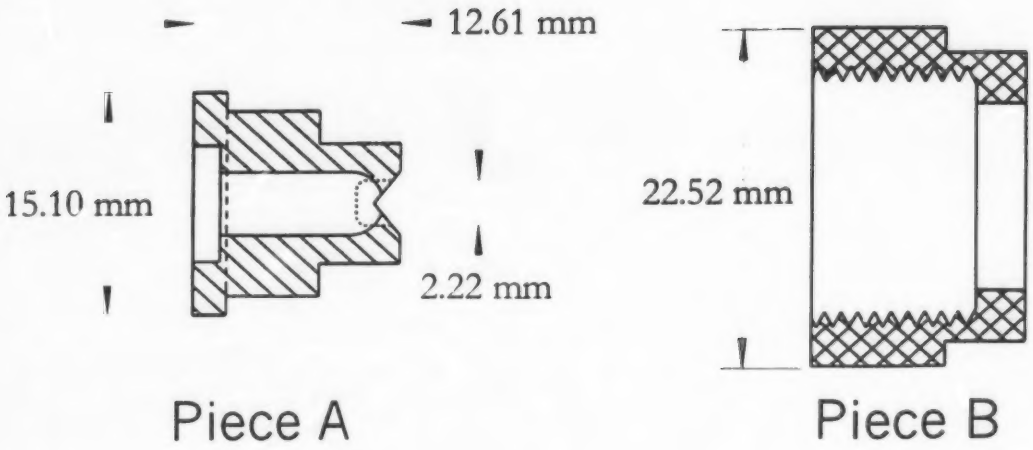
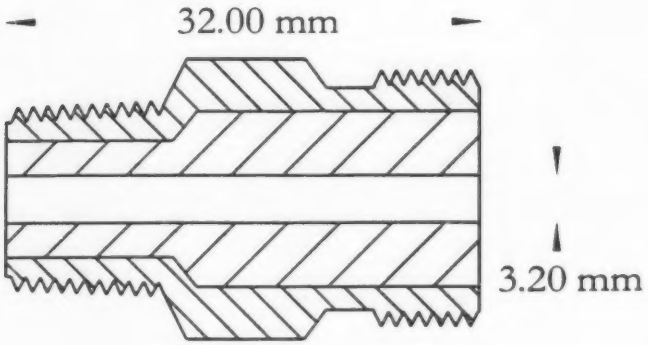


FIGURE 3 – Example of a hydraulic nozzle for test condition RCSW-1





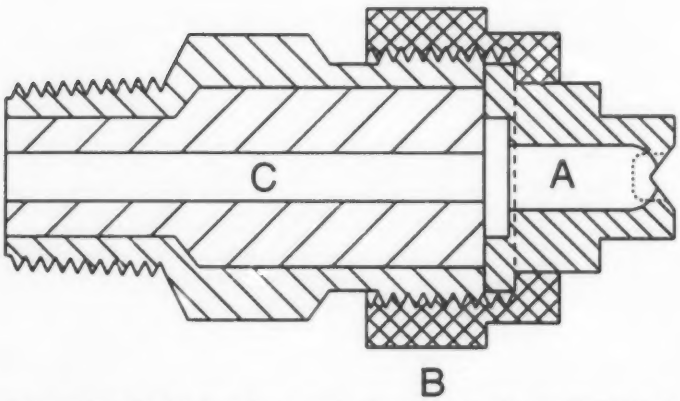


FIGURE 4 – Example of a hydraulic nozzle for test condition RCSW-2

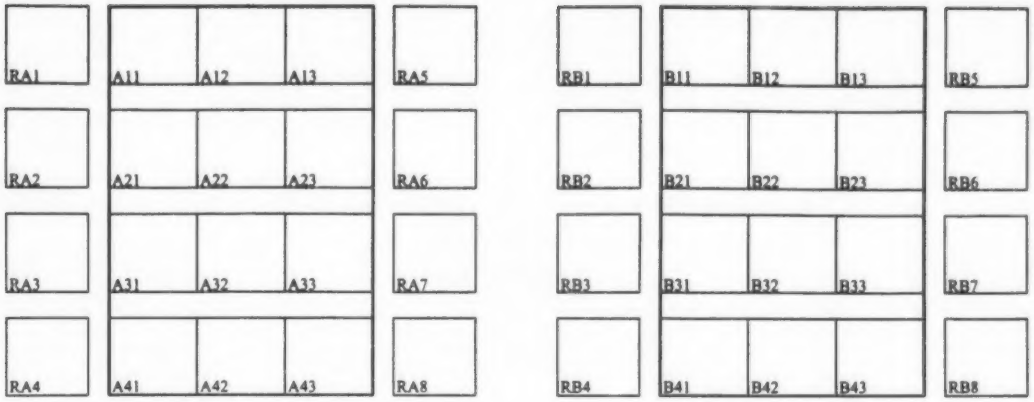


FIGURE 5 – Example of the disposition of reference precipitation catch pans (denoted RA1 to RA8 for plate A and RB1 to RB8 for plate B), each surrounding the 12 precipitation catch pans replacing the test plates (A11 to A43 for test plate A and B11 to B43 for test plate B)

APPENDIX SNW

PROCEDURES FOR ANTI-ICING ENDURANCE TIME TESTING OF AIRCRAFT DEICING/ANTI-ICING FLUID UNDER SNOW CONDITIONS

1. SCOPE

1.1 This document establishes the minimum requirements for test procedures used to carry out anti-icing endurance time tests of aircraft deicing/anti-icing fluids under snow conditions. The primary purpose for such a test method is to determine the snow anti-icing endurance time under controlled laboratory conditions for SAE Type II, Type III, and Type IV fluids.

1.2 While the materials, methods, applications, and processes described or referenced in this appendix may involve the use of hazardous materials, this appendix does not address the hazards that may be involved in such use. It is the sole responsibility of the user to ensure familiarity with the safe and proper use of any hazardous materials and processes, and to take necessary precautionary measures to ensure the health and safety of all personnel involved.

1.3 The values stated in SI units are to be regarded as the standard.

2. REFERENCED DOCUMENTS

2.1 SAE Publications:

Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096-0001.

AMS 4037 Aluminum Alloy Sheet and Plate, 4.4 Cu - 1.5 Mg - 0.6 Mn (2024-T3 Flat Sheet, T351 Plate) Solution Heat Treated

2.2 ASTM Publications:

Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

ASTM D 1193 Reagent Water

2.3 ISO Publication:

Available from International Organization for Standardization, Case Postal #56, rue Varembe, CH-1211, Switzerland.

ISO 10012: Quality assurance requirements for measuring equipment

3. SUMMARY OF TEST

3.1 This test permits the determination of laboratory anti-icing endurance times of SAE Type II, Type III, and Type IV fluids. The test fluids to be evaluated are applied to a test plate exposed to snow conditions. Their anti-icing endurance times are evaluated by measuring the minimum exposure time before a specified degree of freezing occurs. A general description of the tests referred to in this appendix is as follows.

3.2 Snow Simulation Test :

This test involves pouring the fluid onto a test plate inclined at a 10° angle from the horizontal. The anti-icing endurance time is recorded as the time for ice formation to cover 30% of the test plate. Seven tests corresponding to different sets of temperature and snow intensities are produced:

Table 1: Snow Test Conditions

Test Condition	Air Temperature (°C)	Snow Intensity ($g/dm^2/h$)
Snw-A	-3	10
Snw-B	-3	25
Snw-C	-14	10
Snw-D	-14	25
Snw-E	-25	5
Snw-F	-25	10
Snw-G	<-25*	5

**This test will be performed at the lowest usable temperature of the fluid if it is below -25°C.*

It is a fundamental requirement of this test that the simulated snow be in the form of solid crystals.

4 EQUIPMENT AND TEST PARAMETERS

4.1 General:

A description of the minimum requirements for the environmental test chamber and associated test equipment, including test plate and data acquisition, is given below. A summary of the performance requirements for the test equipment is given in Table 2. Any method of collecting and distributing artificial or natural snow is acceptable as long as the snow impinging on the surface of the test plate is in solid form and the resulting anti-icing endurance times meet the anti-icing endurance times of Reference Fluid X (Tables 3 and 4) within a 10% variation (Table 5). Other snow simulation systems that meet the requirements of this appendix are acceptable. Examples of snow simulation systems are described in sections 4.6 and 4.7.

Table 2: Summary of Performance Requirements for Test Equipment

Test Parameters	Requirements
Test chamber	
• Air temperature range	-3°C to at least -25°C
• Average set point temperature	± 0.5°C
• Standard deviation	0.2°C at -3 and -14°C and 0.5°C at -25 and below -25°C
• Minimum temperature sampling rate	1 datum per minute
• Horizontal air velocity	≤ 0.6 m/s
• Relative humidity	> 40%
Test Plate	
• Material	Aluminum alloy AMS 4037
• Surface finish	Average surface roughness: Ra = 0.1 to 0.2 μm
• Test plate dimensions	500 mm long x 300 mm wide x 3.2 mm thick
• Slope	10° ± 0.2
• Temperature at start of test	Within ± 0.5°C of air temperature
Snow distribution apparatus	
• Distance between apparatus and test plate	minimum 500 mm
• Snow intensities	5 g/dm²/h ± 0.5 10 g/dm²/h ± 0.5 25 g/dm²/h ± 0.5
• Snow density	0.25 g/cm³ ± 0.2
• Condition of snow	no evidence of sintering*, agglomeration, or recrystallization

** Sintering may occur 1 to 2 weeks following the collection of the snow, depending on the form of the snow crystals and storage methods.*

Table 3: Formula for Standard Fluid X

650 g	Ethylene Glycol
350 g	Deionized Water
6.0 g	Food Grade Xanthan Gum*

* viscosity: 1200-1600 mPa.s
transmittance: not more than 85%
particle size: 100% through 60 mesh (250 μm) and at least 95% through 80 mesh (180 μm)

Procedure:

1) Pour ethylene glycol into a container;
2) Slowly add the Food Grade Xanthan Gum while mixing;
3) Slowly add water while mixing;
4) Agitate for at least 20 minutes, until a homogeneous mixture is obtained.

Table 4: Fluid X Characteristics

	T (°C)	Value	Tolerance
Refractive Index	20	1.3969	± 0.0006
pH	20	6.56	± 0.04
Viscosity* (mPa.s)	20	40 200	± 21.6%

(Brookfield 0.3 RPM cylindrical spindle # 2, or equivalent)

Table 5: Anti-icing Endurance Times for Standard Fluid X with the Snow Simulation Test

Condition	Temperature (°C)	Icing Intensity ($g/dm^2/h$)	Anti-icing Endurance Time (min)
Snw-A	-3	10	59
Snw-B	-3	25	30
Snw-C	-14	10	35
Snw-D	-14	25	16
Snw-E	-25	5	50
Snw-F	-25	10	25

4.2 Test Chamber (see Table 2):

The plate and air temperature shall be within 0.5°C prior to starting the test. The temperature sensing device shall be in proximity to the test plate, typically within 1.5 m of the side of the test plate, but outside the spray area. The air exchange rate in the chamber shall correspond to an average horizontal air velocity no greater than 0.6 m/s when measured 50 mm above the surface of the test plate. The test chamber shall be equipped with artificial lighting such that it does not interfere with the snow precipitation nor with the air, fluid, and plate temperatures.

The air temperature, plate temperature, and humidity sensing devices shall be linked to an electronic data acquisition system as a means of checking the environmental control characteristics of the test chamber throughout the course of a test run.

4.3 Test Plate (see Table 2):

Each test plate is a removable panel placed on a support housed within the test chamber. Each panel shall be marked with a horizontal line 150 mm from the upper shorter edge of the test plate or with any other permanent pen marking used to estimate the degree of ice formation on fluid-treated panels during the course of a test run.

Each test plate shall be equipped with a temperature sensor located on the underside of the plate. This sensor shall be capable of measuring to an accuracy of ± 0.5°C and shall be linked to an electronic data acquisition system.

The test plate support face shall be inclined at a 10° angle ± 0.2 from horizontal. The test plates shall be placed on the support such that the fluid can freely flow off all edges of the plate. The test stand shall be designed as to minimize the contact between the test surface and support.

4.4 Example of a Snow-Generating System:

4.4.1 General

The snow-generating system described here consists of two steps: artificially making the snow and distributing the artificial snow onto the test plates as clusters of snow.

4.4.2 Artificial Snow-Making:

4.4.2.1 General:

The equipment used for snow-making uses a fine water spray in a high climatic chamber. The spray equipment provides the water spray from a nozzle supplied with low flow water. This equipment is housed in the upper region of the snow-making chamber above a snow collection receptacle. The water conforms to ASTM D 1193, Type IV. The spray equipment is adjusted in order to meet the following criteria:

(a) The supercooled water spray impinges on the surface of the snow collection receptacle and, on contact, forms solid crystals.

(b) The snow has a density of 0.25 g/cm^3 ± 0.02.

(c) The snow is sorted to retain only the agglomerate in the 600 to 1400 µm range.

(d) The snow is preserved in an environment where the temperature is below -10°C. If the snow shows any evidence of sintering, agglomeration, or recrystallization, it is not be used for the snow simulation tests.

The exact type and geometry of the spray system used to generate artificial snow for the test is left to the discretion of the user, provided the foregoing parameters are met.

4.4.2.2 Example of an Artificial Snow-Making Apparatus:

In a cold chamber, the air is cooled to a target temperature of -20°C ± 5. Typical conditions used to achieve the required snow density and grain size include: water flow rate of 70 mL/min, air pressure at 700 kPa, and 2 or 3 hydraulic water spray nozzles. In this example, the water spray nozzle is mounted about 7 m above the snow collection receptacle.

4.4.3 Snow Distribution System:

4.4.3.1 General Requirements:

The snow distribution system shall be able to distribute the snow at rates of 2, 5, 10, and 25 g/dm^2/h in the form of clusters of ice crystals. Each cluster shall have an individual weight of 0.15 g ± 0.05 g or any other weight and distribution pattern such that Reference Fluid X (see Tables 3 and 4) manufactured as described in section 4.1 presents the anti-icing endurance times presented in Table 5. The snow shall be continuously mixed or agitated to avoid clumping, which alters the density of the snow, prior to impinging on the plate.

4.4.3.2 Example of a Snow Distribution System:

The snow used for the tests is contained within a U-shaped aluminum box, 320 mm long, 170 mm high, and 140 mm wide at the top, located above the test plate (Figure 1). The box is suspended from a track 760 mm above the center of the test plate (Figure 2). The track is attached to a motor that provides the lateral movement of the snow box. The lateral displacement speed depends on the desired snow intensity. The snow is continuously stirred inside the box by a rotating system consisting of three blades disposed at 120° angles from each other (Figure 3). Each blade measures 50 mm x 300 mm and consists of a frame housing a mesh. The continuous rotation of the blades prevents clumping of the snow prior to dispensing. The box contains a 20 mm wide opening along the length of the base of the box. This opening houses a 25 mm diameter Teflon cylinder containing 20 cavities arranged in 5 rows of 4 cavities each at 72° spacing (Figure 4). Each cavity has a diameter of 11.2 mm and is drilled to a U-shape. The cavities on each row are spaced at 4.25 mm intervals and out of phase with each other row. The cylinder turns after a given time interval to dispense snow clusters onto the test plate. The rotation speed of the cylinder is predefined to accommodate the desired snowing intensity.

4.5 Temperature Control Equipment:

The air temperature shall be maintained at the required level using heat exchangers connected to temperature control equipment featuring a solid state temperature sensor such as a platinum resistance probe (100 ohms at 0°C) coupled to a proportional temperature controller having a minimum resolution of 0.5°C.

4.6 Air Distribution System:

This system shall consist of a fan or fans to provide air recirculation through the main body of the test chamber and to the heat exchanger. Ducting for the passage of air at both the inlet and the outlet of the heat exchanger shall have entry and exit ports positioned to provide good air recirculation throughout the test chamber. The heat exchanger shall be capable of cooling the air and maintaining it at the specified temperature level. Air flow shall be measured using a suitable anemometer or velometer.

5. CALIBRATION OF TEST EQUIPMENT

5.1 Standard Measuring Devices:

All temperature sensors, humidity sensors, electronic balances, anemometers, and timing devices shall be maintained in a known state of calibration in accordance with recognized international standards such as ISO 10012, by calibrating each measuring device at least once every six months or whenever a piece of equipment is repaired, replaced, or moved, and a written record of the calibrations shall be kept available.

5.2 Average Air Velocity:

The average horizontal component of the air velocity in the test chamber, when measured 50 mm above the centerline at the upper edge of the test plate, must not exceed 0.6 m/s.

5.3 Snow Catch Calibration:

For each anti-icing endurance time test, it is important to establish that even and reproducible snow falls over the surface of the test plates. To carry out this evaluation, ice catch measurements shall be performed under each condition. A summary of the test conditions follows.

Table 6: Snow Intensity Calibration Tests

Condition	Snw-A	Snw-B	Snw-C	Snw-D	Snw-E	Snw-F	Snw-G
Air Temperature (°C)	-3	-3	-14	-14	-25	-25	<-25*
Snow intensity ($g/dm^2/h$)	10 ± 0.5	25 ± 1.0	10 ± 0.5	25 ± 1.0	5 ± 0.2	10 ± 0.5	5 ± 0.2
Standard Deviation ($g/dm^2/h$)	0.5	1.5	0.5	1.5	0.3	0.5	0.3
Calibration Test Duration (min)	30 and 60	30 and 60	15 and 40	10 and 30	10 and 30	10 and 30	10 and 30

** This test will be performed at the lowest usable temperature of the fluid if it is below -25°C.*

To assess the snow catch for each condition, each test plate is replaced with 15 snow catch pans, each 100 mm x 100 mm with a 15 mm edge (see Figure 5). The preweighed pans are weighed upon completion of each test and the difference in the recorded weights is the snow catch for that pan. The snow intensity is the average of the snow catch in the pans and should fall within the values in Table 6. The standard deviation between snow catch pans shall fall within the values in Table 6 for each test condition.

The degree of repeatability shall be checked by performing not less than two successive test runs. The same performance limits must be achieved in each run.

6. TEST PROCEDURE

6.1 Test Plate Cleanliness:

The test plates shall be free of any visible contamination, smears, or stains, except for the horizontal line marked at the 150 mm point from the upper edge of the test plate or any other marking used to estimate ice coverage. Between test runs, any contamination shall be removed by washing with hot water immediately followed by ethanol. If the same fluid is tested on the same plate for two consecutive tests, it is not necessary to clean the plates with ethanol before the second test, hot water is sufficient.

6.2 Prepare 500 mL of the candidate test fluid for each panel to be coated. If more fluid is required, the quantity of fluid actually used shall be mentioned in the report qualification statement. The fluid shall be sheared in accordance with the specification for the respective SAE Type fluid. The test shall start within two hours after the product has been sheared, but not within the first 20 minutes after shearing.

6.3 Ensure the test chamber is at the required temperature. Verify that the test plate temperature is within 0.5°C of the ambient air temperature. Fluids shall be applied at 20°C ± 5.

6.4 Pour the fluid onto each test plate, start the timing device, cover each test plate with a removable cover, and turn on the snow distribution system. After 5 minutes remove the cover from each test plate. Observe the panels and, when failure occurs as described in section 6.5, record this time as the anti-icing endurance time. When the snow distribution system is turned off, estimate the snow catch for each test panel. If the snow catch is within the specified limits for the test being conducted, the time recorded is valid for the snow simulation test. With the system described in section 4.4.3, the box must be weighed before and after each test.

6.5 Failure criterion:

Failure is called when the accumulating precipitation fails to be absorbed over 30% of the panel. Pen marks on the plate can be used to estimate the area of failure. For instance, a line drawn across the plate at 150 mm from the top edge will delineate an area corresponding to 30% of the plate.

6.6 Reproducibility/Precision:

The snow simulation test is dynamic by nature, and small variations can be expected. Each test condition shall be tested on at least two panels. If the maximum variation of the anti-icing endurance time is less than 10% of the average anti-icing endurance time, report this time. If not, repeat testing on two additional panels, for a total of four data points. The highest and lowest points shall be discarded, and the average of the two remaining values shall be the anti-icing endurance time for that condition.

6.7 Report:

The report shall state the name and address of the facility conducting the tests, together with a statement confirming the test facility is autonomous of the manufacturer or vendor of the fluid. The following information shall also appear on the test document:

Date tests conducted
Manufacturer or vendor's name and address
Name or reference number and lot number of the fluid tested
Type of fluid (SAE Type II, SAE Type III, or SAE Type IV)
Summary of test results, and calibration results for each test
Method of estimation of failure area

Condition of fluid: Concentrate as supplied to test facility, or diluted with hard water as defined in the specification and subsequently sheared in accordance with specification giving the dilution as a ratio (example: SAE Type II diluted 75:25 with hard water).

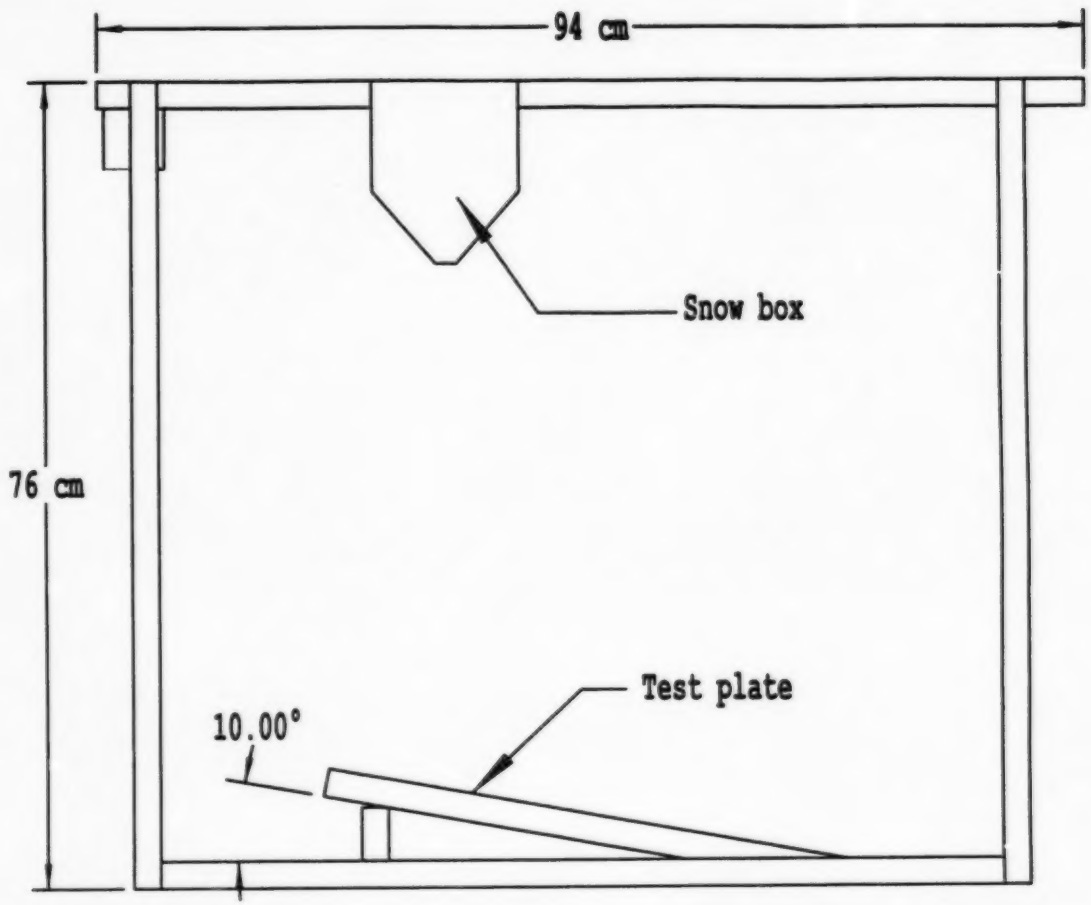


FIGURE 1 – Snow box in support above test plate

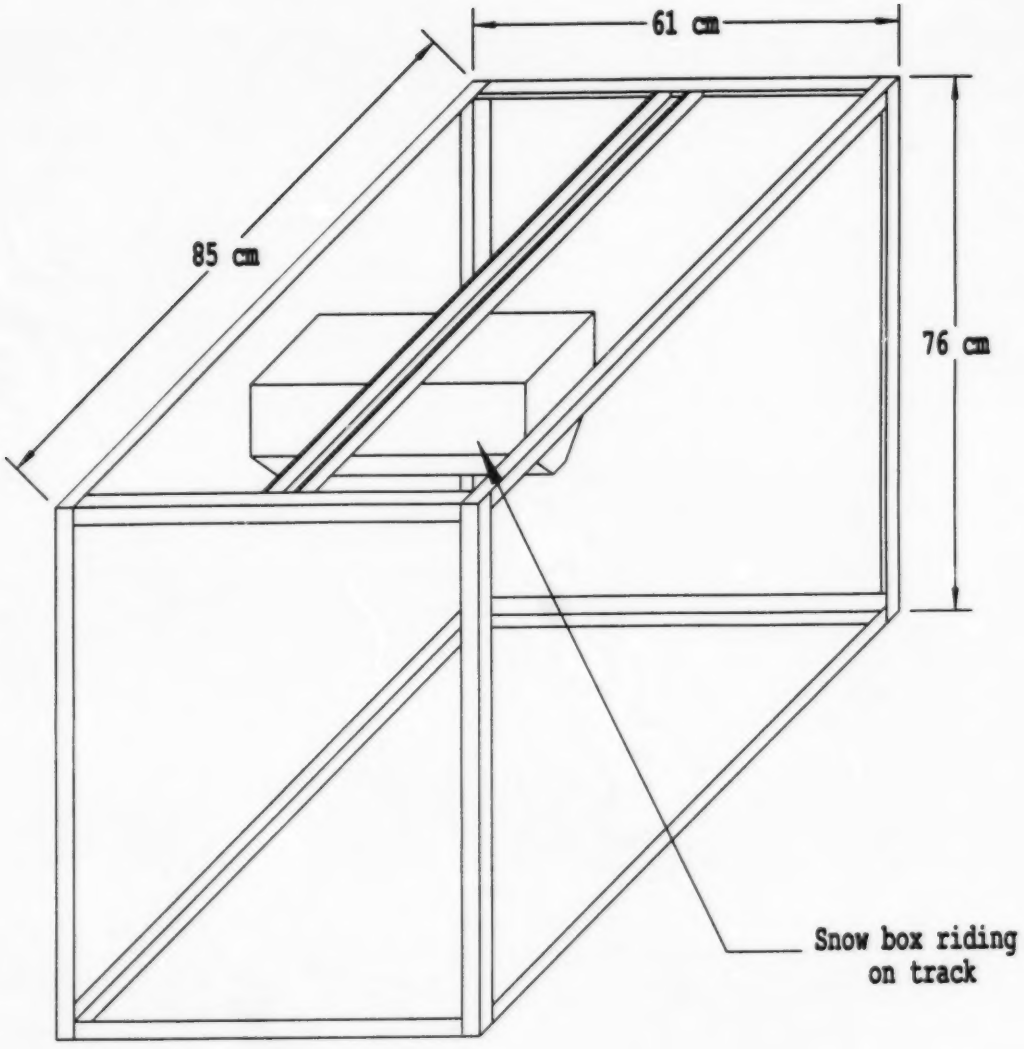


FIGURE 2 – Snow box on track of support

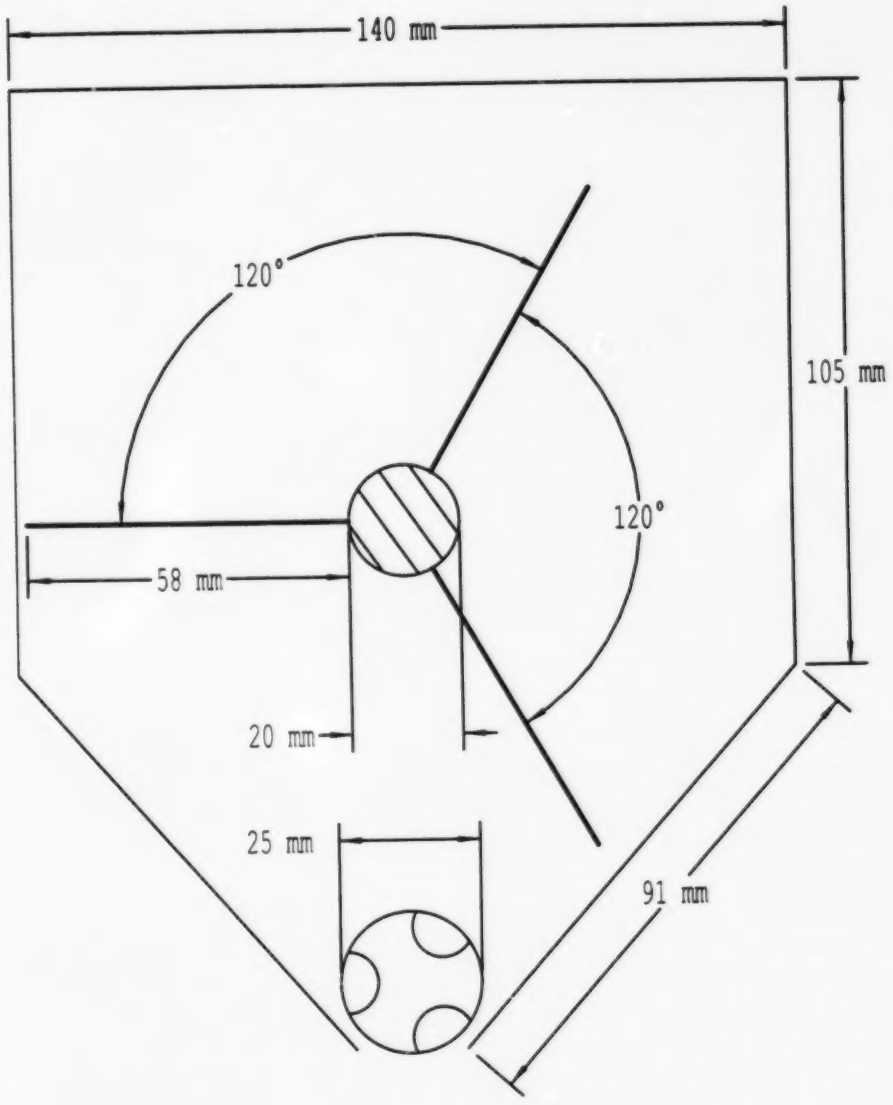
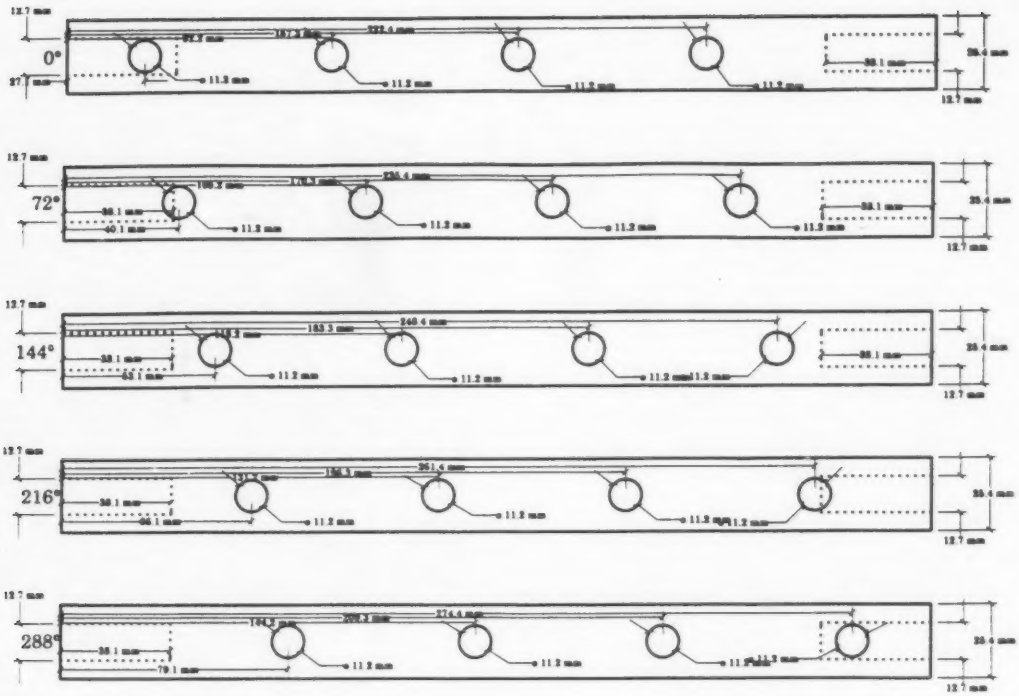


FIGURE 3 – Cross-section of snow box



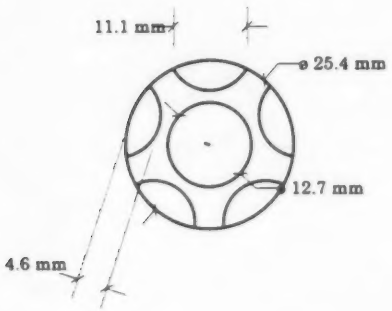


FIGURE 4 – Side views and cross-section of Teflon cylinder with 5 rows of 4 cavities that transfer snow from box to test plate

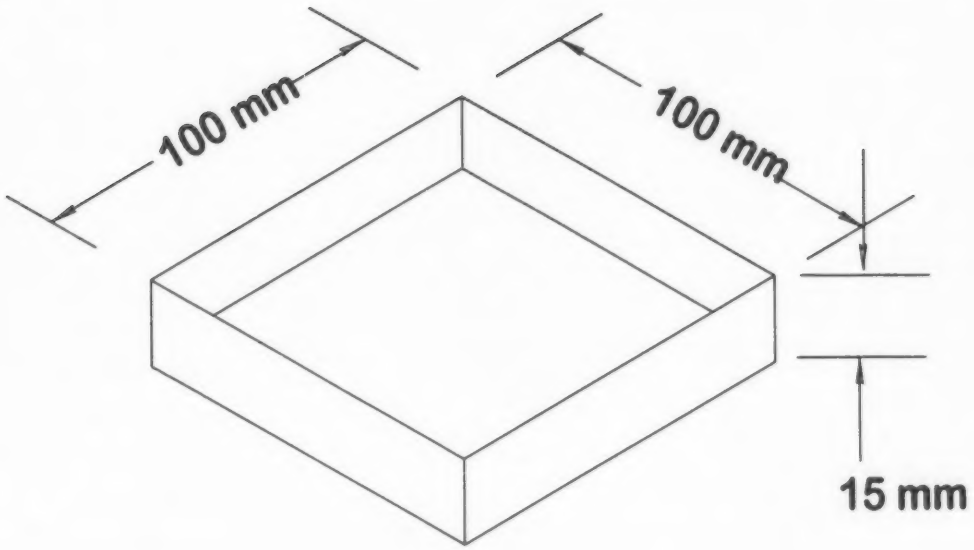


FIGURE 5 – Snow catch pan